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A LOW TEMPERATURE PRECISION GAS THERMOMETER

BY

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A THESIS

SUBMITTED TO THE FACULTY OF GRADUATE STUDIES IN
PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE
DEGREE OF MASTER OF SCIENCE

DEPARTMENT OF PHYSICS

EDMONTON, ALBERTA

FEBRUARY, 1966

ABSTRACT

For the realization of the absolute temperature scale in the range 4-20°K a constant volume gas thermometer with accessories (cryostat, manometer and gas filling system) was designed. The gas thermometer was calibrated against the ⁴He vapour pressure scale of temperatures¹⁾.

Two resistance thermometers - a carbon thermometer and a germanium thermometer - were calibrated against this gas thermometer in the temperature range 4° to 20°K. The reproducibility of the carbon thermometer at about 4° is $\pm 0.003^{\circ}\text{K}$ and that of the germanium thermometer $\pm 0.001^{\circ}\text{K}$. The interpolation formula for deriving the temperature from a given resistance value and the deviation of the interpolated temperature from true temperature are discussed. A discussion of the observed temperature dependence of electrical resistance for the doped semiconductor thermometer is also given.

The temperature of the superconducting transition of high purity lead has been determined by the gas thermometer to be $7.196^{\circ}\text{K} \pm 0.005$. The width of the transition zone is 0.003°K . This suggests that the superconducting transition temperature of lead may be used as a secondary fixed point.

ACKNOWLEDGEMENTS

I wish to express my gratitude to Dr. J.P. Franck, my research Supervisor, for suggesting this project and for his able guidance throughout the course of this research.

I also wish to thank Mr. Rene Wanner for his assistance during some of the runs. Thanks are due to Miss J.G. Seale for writing the computer programme for some of the calculations.

The assistance of the technical staff of the Physics Department is greatly appreciated. The assistance of Miss Sharon Page in the preparation of the manuscript is also acknowledged..

Finally it is a pleasure to acknowledge the financial support of the Physics Department and the National Research Council, Ottawa.

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INTRODUCTION

The secondary thermometers for measurements at low temperatures are thermocouple, resistance thermometer, vapour pressure thermometer and magnetic thermometer. The secondary thermometer is required to be calibrated by a primary standard at a few points in the desired temperature range.

In the temperature range of interest in this work (4° to 20°K) carbon thermometers have been successfully used. They have a negative temperature coefficient of resistance. A decrease in temperature results in enhanced sensitivity which is the situation opposite to that of a metallic thermometer (e.g. platinum resistance thermometer). An important short-coming of carbon thermometers is their limited reproducibility on thermal cycling to room temperature.

The development of semiconductor thermometers was stimulated by the obvious need of a thermometer in the range below 20°K which is more reproducible than the carbon thermometer. Semiconductor thermometers have resistance - temperature characteristics which are somewhat similar to that of the carbon thermometer apart from the additional advantage of high reproducibility.

To set up the facilities for calibration of secondary thermometers a constant volume gas thermometer was designed.

A carbon resistance thermometer and a semiconductor thermometer were calibrated in the range 4° to 20°K by this gas thermometer. Temperatures derived from interpolation formulae were compared with true temperatures. Reproducibility tests on both thermometers were carried out over a period of about three months.

The results of measurement of the superconducting transition temperature of high purity lead supplied to us by Consolidated Mining and Smelting Co. of Canada suggest this temperature as a secondary fixed point.

TEMPERATURE MEASUREMENT2.1 Introduction

The definition of the thermodynamic temperature scale is tied to the second law of thermodynamics. Two corollaries of the second law are:

If Q_1 and Q_2 are the quantities of heat flowing to a reversible heat engine operating on a Carnot cycle between two heat reservoirs at temperatures t_1 and t_2 respectively, then

a) Q_1 and Q_2 cannot have the same algebraic sign,

$$(2.1)..b) \quad \frac{Q_1}{Q_2} = \frac{-f(t_1)}{f(t_2)} = g(t_1, t_2).$$

The fact that $g(t)$ is a universal function of temperature allows one to use Eq. (2.1) as the definition of a temperature scale. If we define the temperature scale by setting $f(t) = T$, then

$$(2.2)... \quad \frac{T_1}{T_2} = - \frac{Q_1}{Q_2}.$$

This is the definition of the absolute thermodynamic temperature scale, and is independent of the working substance used in the thermometer.

The temperature scale based on an ideal gas is the same as the thermodynamic scale except for the arbitrary choice of size of the degree.. It is this reason that makes gas thermometry so important. An ideal gas is defined by two properties:

- a) Boyle's law: which states that the pressure-volume product of a definite mass of a gas is constant at a definite temperature T ,

$$(2.3) \quad \dots \quad PV = f(T).$$

- b) Joule's law: the internal energy of an ideal gas is independent of the volume, dependent only on the temperature,

$$(2.4) \quad \dots \quad \left(\frac{\partial U}{\partial V} \right)_T = 0.$$

No real gas satisfies these requirements exactly, but an ideal gas can be approximated as closely as desired by reducing the pressure.

Since PV is a function of temperature alone, we can define the ideal gas temperature $\mathcal{T}$ by

$$(2.5) \quad \dots \quad \frac{(PV)_{\mathcal{T}}}{(PV)_{\mathcal{T}_2}} = \frac{\mathcal{T}}{\mathcal{T}_2}.$$

where $\mathcal{T}_2$ is the defining fixed point.

$$\text{or} \quad (PV)_{\mathcal{T}} = \mathcal{T} ((PV)_2 / \mathcal{T}_2)$$

$$(2.6) \quad \dots \quad = A \mathcal{T}$$

The constant A must be determined experimentally, but under conditions that the gas at hand satisfies the ideal gas laws.

Next, to show that the thermodynamic temperature scale as defined by Eq. (2.2) is identical with the ideal gas scale defined above, we consider the thermodynamic relation:

$$(2.7) \quad \dots \quad P = T \left(\frac{\partial P}{\partial T} \right)_V - \left(\frac{\partial U}{\partial V} \right)_T$$

where U is the internal energy and T is the thermodynamic temperature as defined by the second law.

For an ideal gas Eq. (2.7) becomes

$$(2.8) \quad \dots \quad P = T \left(\frac{\partial P}{\partial T} \right)_V$$

Differentiating the ideal gas equation (at constant volume)

$$PV = A\mathcal{J}$$

$$V\left(\frac{\partial P}{\partial \mathcal{J}}\right)_V = A = \frac{PV}{\mathcal{J}}$$

$$(2.9) \quad \dots \quad \text{or} \quad P = \mathcal{J} \left(\frac{\partial P}{\partial \mathcal{J}}\right)_V$$

Comparing (2.8) and (2.9)

$$\frac{T}{\mathcal{J}} = \left(\frac{\partial T}{\partial \mathcal{J}}\right)_V$$

or

$$\left(\frac{\partial \ln T}{\partial \ln \mathcal{J}}\right)_V = 1$$

Therefore

$$\ln \mathcal{J} = \ln T + \alpha(V)$$

where the constant α does not depend on temperature. This gives

$$(2.10) \quad \dots \quad \mathcal{J} = \text{const. } T = \beta(V) T.$$

Therefore for a gas at constant volume $\mathcal{J}$ is proportional to T .

$$\begin{aligned} \text{As } PV &= A \mathcal{J} \\ (2.11) \quad . . . &= A \cdot \beta(V) \cdot T \end{aligned}$$

for Boyle's law to hold, we must have $\beta(V)$ to be independent of volume. Thus β is independent of volume and temperature, meaning thereby that the two scales are proportional. Since the size of the degree is a matter of choice, the two scales are chosen to be identical.

2.2. Realization of Thermodynamic Temperature Scale with Gas Thermometer

The basic point in gas thermometry is to devise a method in which a real gas can be made to behave like an ideal gas or if it is not possible, to devise a method where the differences in behaviour between the real and ideal gases are known and can be corrected for.

Two different types of gas thermometers are:

- a) Constant volume gas thermometer - in which one keeps the volume of gas constant and measures pressure as a function of temperature.
- b) Constant pressure gas thermometer - in which one keeps the pressure of gas constant and measures the volume as a function of temperature.

Gas thermometry not only gives results directly in terms of absolute temperature and has a sensitivity²⁵⁾ which can be increased by using higher filling pressures at lower temperatures but is unaffected by magnetic fields and is easily adapted to differential thermometry for measurement of small temperature differences.

2.3 Equation of State of a Real Gas

The thermometric fluid of a gas thermometer is not a perfect but a real gas. For any real gas the ideal behaviour is approached only as the pressure is made infinitely small i.e. the density tends to zero. The equation of state of a real gas is

$$(2.12) \quad PV = RT \left[1 + \frac{B}{V} + \frac{C}{V^2} + \dots \right]$$

where B, C,..... are called virial coefficients and are functions of temperature, but not of volume or pressure.

2.4 Gas Thermometry

In the constant volume gas thermometer a definite mass of gas is enclosed in a bulb of invariant volume. The pressure of gas is measured while the bulb is thermostated

at the respective temperature. The pressure is usually measured on some manometer at room temperature and is connected to the bulb by a fine capillary. Thus a part of the system is at a temperature different from that of the bulb. The volume of gas outside the bulb constitutes the dead volume. For a given quantity of gas

$$P \sum_i \frac{v_i}{T_i}$$

is constant, where the v_i is the volume at the temperature T_i . If v_b is the volume of the bulb and v is the dead volume assumed to be at a constant temperature T_o , P_o is the pressure at temperature T_o , then (neglecting the volume of the capillary with a changing temperature distribution) for an ideal gas,

$$P_o(v_b + v)/T_o = P\left(\frac{v_b}{T} + \frac{v}{T_o}\right)$$

where P is gas pressure corresponding to temperature T of bulb,

$$(2.13) \quad T = T_o(P/P_o) \bigg/ \left(1 + \frac{v}{v_b} (1 - P/P_o)\right).$$

The sensitivity of the thermometer, $\frac{dP}{dT}$, is

$$(2.14) \quad \dots \quad \frac{dP}{dT} = \frac{P_0}{T_0} (1 + \frac{v}{v_b} (1 - P/P_0))^2 \bigg/ (1 + \frac{v}{v_b}).$$

Thus the sensitivity depends on the initial filling pressure and temperature and on the relative magnitudes of v_b and v . If v is negligible,

$$(2.15) \quad \dots \quad \frac{dP}{dT} = \frac{P_0}{T_0}.$$

If v is not negligible, its effect is to decrease the sensitivity in the region T_0 by a factor $(1 + \frac{v}{v_b})$,

$$(2.16) \quad \dots \quad \left(\frac{dP}{dT}\right)_{T=T_0} = \frac{P_0}{T_0} \bigg/ (1 + \frac{v}{v_b}).$$

At very low temperatures the influence of dead volume is to increase the sensitivity by the same factor $(1 + \frac{v}{v_b})$, viz.

$$(2.17) \quad \dots \quad \frac{dP}{dT} = \frac{P_0}{T_0} (1 + \frac{v}{v_b}).$$

This equation shows that a gas thermometer with relatively large dead volume³⁾ has a large sensitivity at low temperatures. However if a linear dependence of P on T is required so that

the sensitivity is more or less independent of temperature then the volume of the bulb should be large in comparison with the dead volume, Eq. (2.15). This means physically that the major part of the gas is in the bulb at all temperatures and therefore to a first approximation P is proportional to T . In making corrections for the volume of gas in the capillary the temperature gradient of the capillary must be known accurately.

As said above the sensitivity depends on the initial filling pressure and temperature, but a large initial filling pressure may result in the condensation at sufficiently low temperatures and high non-ideality corrections.

2.5 Vapour Pressure Thermometry

The secondary instruments that have been most practical and reproducible for measurements at low temperatures are the thermocouple, the resistance thermometer, the magnetic thermometer and the vapour pressure thermometer. By definition a secondary standard is a thermometer which can be calibrated in a standardizing laboratory at a few points in the desired range and then can be trusted to retain its calibration and serve as a working thermometer throughout the range with an accuracy comparable with that of the

calibration points.

The vapour pressure thermometer is not a suitable instrument for general use because of its limited temperature range, but it is valuable for secondary calibrations since it can yield temperatures throughout its range which are, in effect, a continuous series of secondary fixed points.

Over the temperature range 0.25 to 5.2°K, 14 to 20°K, 55 to 90°K and 63 to 77°K the vapour pressures of liquid ^3He and ^4He , hydrogen, oxygen and nitrogen can be used. ~~for calibrating the resistance thermometer.~~

A formula connecting the vapour pressure and temperature can be obtained by integrating the Clausius Clapeyron equation:

$$(2.18) \quad \frac{dP}{dT} = \frac{L}{T(V_g - V_l)}$$

where L is the heat of vaporization of liquid, V_l and V_g are molar volumes of the liquid and the gas respectively.

Writing $PV_g = RT(1 + \frac{B}{V})$

$$L = L_0 + a'T$$

Expressing B in Amagat units

$$(2.19) \quad \dots \quad 10^3 B = 0.6824 - 17.244 T^{-1} \quad \text{for helium}^{19)}$$

and neglecting V_ℓ , Eq. (2.18) becomes

$$(2.20) \quad \dots \quad \frac{d \log P}{dT} = \frac{C_1}{T} + \frac{C_2}{T^2} + \frac{C_3}{T^3}.$$

Integrating,

$$(2.21) \quad \dots \quad \log P = \frac{a}{T} + c_1 \log T + \frac{c}{T^2} + d$$

where a , b , c , d are constants. Although equations of this type can be fitted closely to the experimental values of vapour pressure, it is more convenient to tabulate P as a function of T obtained from primary standards and use such a table to find temperatures from experimental values of vapour pressures. A trace of impurity in the liquid will change the vapour pressure of the liquid at a given temperature and so calls for the use of pure liquid. In the case of helium all impurities will be frozen out. It is, however, important to use isotopically pure helium since the vapour pressures of ^3He and ^4He are substantially different.

The vapour pressure thermometer operates on the principle that when a liquid and its vapour are in thermal equilibrium, the pressure depends only on the temp-

erature. Typically, it consists of a bulb containing a liquefied gas, a pressure transmitting tube and a mechanism for pressure measurement. The bulb of the thermometer must always contain both liquid and vapour, whereas the capillary and the manometer contain only vapour. Since there is no dead space correction, a wider connecting tube can be used so that the thermomolecular pressure difference may be negligible. However, precautions against heat flow down the tube must be taken.

For the measurement of temperature the vapour pressure bulb is held in thermal contact with the substance under study. Because the indicated pressure corresponds to the temperature of the coldest point in the system it is necessary that no part of the tube be at a temperature lower than that of the vapour pressure bulb. To eliminate the possibility of a cold spot developing, sometimes shielding of the vapour pressure tube by a vacuum jacket is done.

2.6 Helium Vapour Pressure Scale of Temperatures

In low temperature physics where the basic cooling is produced by liquid helium it is very convenient to use the helium vapour pressure as a thermometric indicator. The vapour pressure-temperature relation for saturated vapour pressure has been deduced in the past from measurements with gas thermometers. The 1958 ^4He vapour pressure scale of temperatures¹⁾ was approved as an international standard for thermometry from 1° to 5.2°K by the International Committee on Weights and Measures at a meeting in October 1958.

At temperatures below 2.2°K liquid ^4He undergoes a transformation to a liquid state called the superfluid state, He II. The superfluid state causes difficulty in accurate vapour pressure thermometry because of the superfluid or creeping film which covers all solid surfaces in contact with the superfluid liquid. ^4He is transported through this film to warmer portions of the apparatus where the film is vaporized. - The vapour then recondenses at the surface of the liquid resulting in a large heat influx to the liquid in a ^4He vapour pressure bulb. This heat flux can cause the liquid ^4He to be hotter than the

walls of its container. This film reflux causes errors in ^4He thermometry. The light isotope of helium, ^3He , has a much higher vapour pressure (120 and 8600 microns of Hg at 1°K for ^4He and ^3He respectively) and the liquid does not become superfluid down to at least 0.007°K . Hence ^3He vapour pressures are practical for use in thermometry at temperatures where ^4He would cease to provide a reliable temperature. Roberts, Sherman and Sydoriak⁴⁾ have published a ^3He vapour pressure scale of temperatures.

The conditions necessary for determining the temperature of the object by measurement of vapour pressure of liquid are:

- a) there must be thermal equilibrium between object and liquid,
- b) the pressure at which the liquid is in thermal equilibrium with its saturated vapour must be determined accurately and manometer corrections be applied,
- c) cold spots must be avoided. The pressure transmitting tube may be wound with an electric heater or vacuum jacketed if it appears likely that cold spots exist at a lower temperature than the condensed liquid in the bulb.

2.7 Carbon Resistance Thermometers

In many experiments at low temperatures it is necessary to have a sensitive and reproducible secondary thermometer with low heat capacity.

In the region below about 20°K, metallic resistance thermometers become quite insensitive and resistance thermometers made of carbon or certain semiconductors become the most useful secondary thermometers. Carbon thermometers possess the following characteristics:

- 1) high thermometric sensitivity,
- 2) low magnetic field sensitivity,
- 3) low sensitivity to measuring current,
- 4) low heat capacity,
- 5) reasonably good reproducibility,
- 6) convenient to use.

As the resistance of carbon thermometers varies approximately exponentially with temperature and has a negative temperature coefficient, $(\frac{1}{R} \frac{dR}{dT})$, of resistance, a decrease in temperature results in enhanced sensitivity and a larger resistance. The rapid variation of resistance with temperature does, however, limit the useful range of a given resistor.

Carbon thermometry was first introduced by Giaque in 1938. In 1950 Clement and Quinnel^{5,6)} studied the low temperature behaviour of several types of commercial carbon composition radio resistors in an effort to find sensitive and reproducible thermometers for low temperature work. The resistors manufactured by the Allen Bradley Co. were found to exhibit consistently high sensitivity and reproducibility. They developed a semi-empirical formula relating temperature and resistance,

$$(2.22) \quad \log R + K/\log R = 2B + A/T,$$

where K, A and B are constants to be determined experimentally. The reproducibility of calculated temperatures upon cycling between liquid helium and room temperatures was within $\pm 0.1\%$. The constants in this equation are given by

$$2B \pm 3\% = 1.62 \log R_{290} + 0.27$$

$$A \pm 9\% = 1.60 \log R_{290} + 0.48$$

$$(2.23) \quad K \pm 6\% = 0.594 (\log R_{290})^2 + 0.377 \log R_{290} - 0.121$$

where R_{290} is the resistance in ohms at room temperature. Since $K = B^2$ within the limits of accuracy, the empirical formula can be approximated by:

$$(2.24) \quad \text{or} \quad \left[\frac{\log R}{T} \right]^{\frac{1}{2}} = a + b \log R.$$

The method of manufacture²⁾ of carbon resistors is a major factor in determining their suitability for thermometric work. The superior reliability of Allen Bradley resistors presumably results from the intimate bonding of terminals to carbon and the forming technique used in their manufacture.

Brown, Zemansky and Boorse⁷⁾ using Allen Bradley resistors described their calibration curves in the range 2 to 20°K by the empirical formula:

$$(2.25) \quad \log R = A + BT^{-1} + CT^{-2} - DT^2.$$

Morrison, Patterson and Dugdale⁸⁾ compared the interpolation formulae (2.22 and 2.25) using 10 ohms, $\frac{1}{2}$ W, carbon thermometers and concluded the superiority of formula (2.22) over (2.25). They found that formula (2.25) gives too large a deviation of the carbon thermometer scale from the thermodynamic scale at low temperatures (deviation is 0.23°K at 1.7°K as compared to 0.06°K when formula (2.22) is used).

2.8 Semiconductor Thermometers

Although the carbon thermometer is a sensitive secondary thermometer below 20°K , it suffers from the short-coming that the R-T characteristic is not very stable. The stability can be improved by careful protection against thermal or mechanical shocks.

The development of semiconductor thermometer was stimulated by the obvious need of a thermometer in the low temperature range whose electrical characteristics are unaffected by conditions encountered during normal use over a period of years.

The role of semiconductors as thermometers was reviewed by Friedberg in 1954. Estermann, Friedberg⁹⁾ and others found that certain impurity concentrations in germanium gave R-T characteristics which were desirable but encountered difficulties with regard to contacts, reproducibility and surface cracking.

In 1951 Friedberg⁹⁾ investigated germanium thermometers and reported their reproducibility to be about $\pm 0.001^{\circ}\text{K}$ at liquid helium temperature on thermal cycling between room temperature and liquid helium temperature. In 1962 Kunzler, Geballe and Hull²⁴⁾ fabricated germanium thermometers which were reproducible to about $\pm 0.0001^{\circ}\text{K}$.

Edlow and Plumb¹⁰⁾ found that germanium thermometers fabricated by Texas Instrument Company were inferior to carbon thermometers in reproducibility.

A restriction on the applicability of semiconductor thermometers is the need for calibration against a primary thermometer since no simple mathematical formula fits the data over the temperature range. Thus it is not possible to have a semiconductor thermometer of which we measure the resistance at one or two fixed points and then have a ready-made calibration by reference to a standard function or a numerical table of values.

Germanium semiconductor thermometers seem to offer a promise as a low temperature thermometer with high sensitivity and good reproducibility by improved fabrication techniques.

2.9 Impurity Conduction in N-Doped Semiconductors

Semiconductors differ from metals in having high resistance and large negative temperature coefficient of resistance. The large temperature coefficient suggests their usefulness as secondary thermometers.

The resistance-temperature characteristic of a semiconductor can be divided into four regions:

- a) intrinsic conduction region,
- b) extrinsic exhaustion region,
- c) extrinsic conduction region,
- d) impurity conduction region.

The conductivity of intrinsic semiconductor arises from the contribution of both electrons and holes. If n_e and n_h are electron and hole densities in the conduction and valence bands respectively and μ_e , μ_h are their mobilities, then the conductivity of material in the intrinsic conduction region is given by

$$(2.27) \quad \sigma = n_e e \mu_e + n_h e \mu_h \\ = \text{const.} \cdot e^{-\alpha/T}$$

where α is a constant of the semiconductor. The conductivity of a semiconductor in the low temperature range can be represented empirically¹¹⁾ by

$$(2.28) \quad \sigma = \frac{1}{\rho} = c_1 \exp(-E_1/KT) + c_2 \exp(-E_2/KT) \\ + c_3 \exp(-E_3/KT)$$

The first exponential term represents the extrinsic conductivity with donor ionization energy E_1 . The second term refers to the middle portion of the plot of $\log \rho$

vs. $1/T$. The last term is the lowest temperature portion of conductivity. E_1 and c_1 can be determined from $\log \rho$ vs. $1/T$ graph. E_3 corresponds to the slope at lowest temperatures and is much smaller than E_1 . The second term is found only in samples with impurity content greater than $10^{16}/\text{c.c.}$. When the impurity is smaller than $10^{16}/\text{c.c.}$ this term is much smaller than the first.

To account for the low temperature behavior of Ge, Hung¹²⁾ postulated that the impurity states interact and form an impurity conduction band. In such a case, an electron has the possibility of moving from one impurity state to another in its neighborhood, so that the states are no longer localized and conduction in the impurity band takes place. When simultaneous conduction in the conduction band and impurity band is considered, the conductivity is given by

$$(2.29) \quad \sigma = n_c e \mu_c + n_i e \mu_i$$

where n_c and μ_c are concentration and mobility of electrons in conduction band, whereas n_i and μ_i refer to the donor band. At higher temperatures conduction in impurity band

can be neglected and

$$(2.30) \quad \sigma = n_c e \mu_c.$$

With decreasing temperature n_c decreases because there is not sufficient thermal energy available to excite electrons into conduction band. In the limit of low temperature, conduction in the conduction band can be neglected and

$$(2.31) \quad \sigma = n_i e \mu_i.$$

Equations (2.29, 2.30 and 2.31) give the conductivity at intermediate, high and low temperatures respectively.

This impurity band model cannot explain the impurity conduction in lightly doped semiconductors because the formation of a band is not possible. Blakemore¹³⁾ pointed out that in a sample of weakly interacting impurities, conduction at low temperatures is possible only if the compensating p-type impurities have been added to n-type sample. The compensation, therefore, plays an essential role in the mechanism of conduction. This view of impurity conduction at low impurity densities is as follows:

When donor impurities are partly compensated by acceptor impurities the latter become negative ions by

acquiring electrons from the former. If there are N_d donors and N_a acceptors with $N_d > N_a$, N_a electrons will be transferred from the donors to the acceptors. This leaves $(N_d - N_a)$ electrons distributed among N_d donors to give rise to conduction. The electrons jump from one impurity to another. This impurity conduction process is rather feeble, in that the hopping of the electrons from one impurity atom to other is rather sluggish. A small thermal energy is required to make electrons jump from donor to donor and conductivity in impurity conduction range is of the form $c_3 \exp(-E_3/KT)$. The quantities c_3 and E_3 depend on N_d and N_a . The acceptors themselves do not contribute to impurity conduction in n-type material, because they are filled with electrons from the donors. Of course, the opportunities for conduction decrease again if N_a becomes almost as large as N_d and this has been confirmed experimentally¹³⁾.

A small addition of compensators in lightly doped samples decreases the low temperature resistivity because the number of empty majority centres is increased. On the other hand the addition of compensators to heavily doped samples increases the resistivity because the addition

of compensators decreases the number of carriers participating in impurity conduction and the mobility also decreases. The fact that an increase in compensation has opposite affect on impurity conduction in the high and low impurity concentration ranges suggest this as a valuable tool for distinguishing the two types of impurity conduction processes. The impurity conduction process accounts for the finite resistance at low temperature and makes germanium thermometers usable to quite low temperatures.

APPARATUS3.1 Description

The Cryostat shown in Fig. 3.1 was designed and assembled in this laboratory. It is composed of the following principal parts:

1. Gas thermometer bulb (1) to which the carbon thermometer (25), semiconductor thermometer (26) and the high purity lead sample (23) for finding its superconducting transition temperature are thermally bonded. The vapour pressure chamber (32) soldered to the bulb is used for calibration of gas thermometer.
2. Adiabatic Shield (3) surrounding the bulb is used to minimize heat leaks.
3. Experimental jacket (4) enclosing the adiabatic shield is connected through (5) to a high vacuum system for evacuation to minimize gaseous conduction.
4. Outer Jacket (15) enclosing the above and the liquid air and helium containers (13 and 7) is connected to another high vacuum system (16).
5. Electrical wiring of cryostat and circuits for controlling the temperature and measurement of resistance.

FIGURE 3.1 a

DIAGRAM
OF THE
APPARATUS

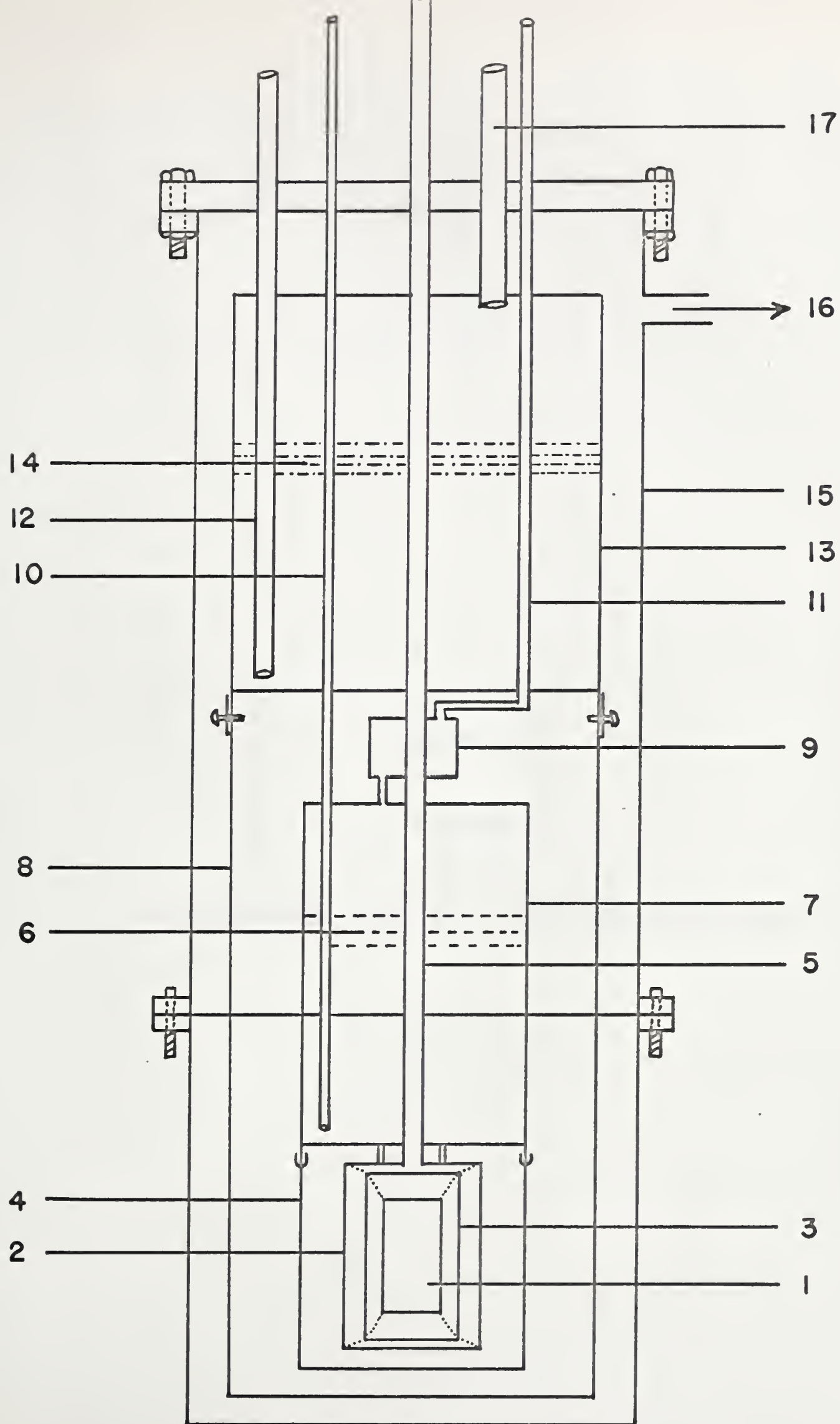


FIG. 3. 10.



FIGURE 3.1 b

DIAGRAM

OF THE

GAS THERMOMETER BULB AND VAPOUR PRESSURE CHAMBER

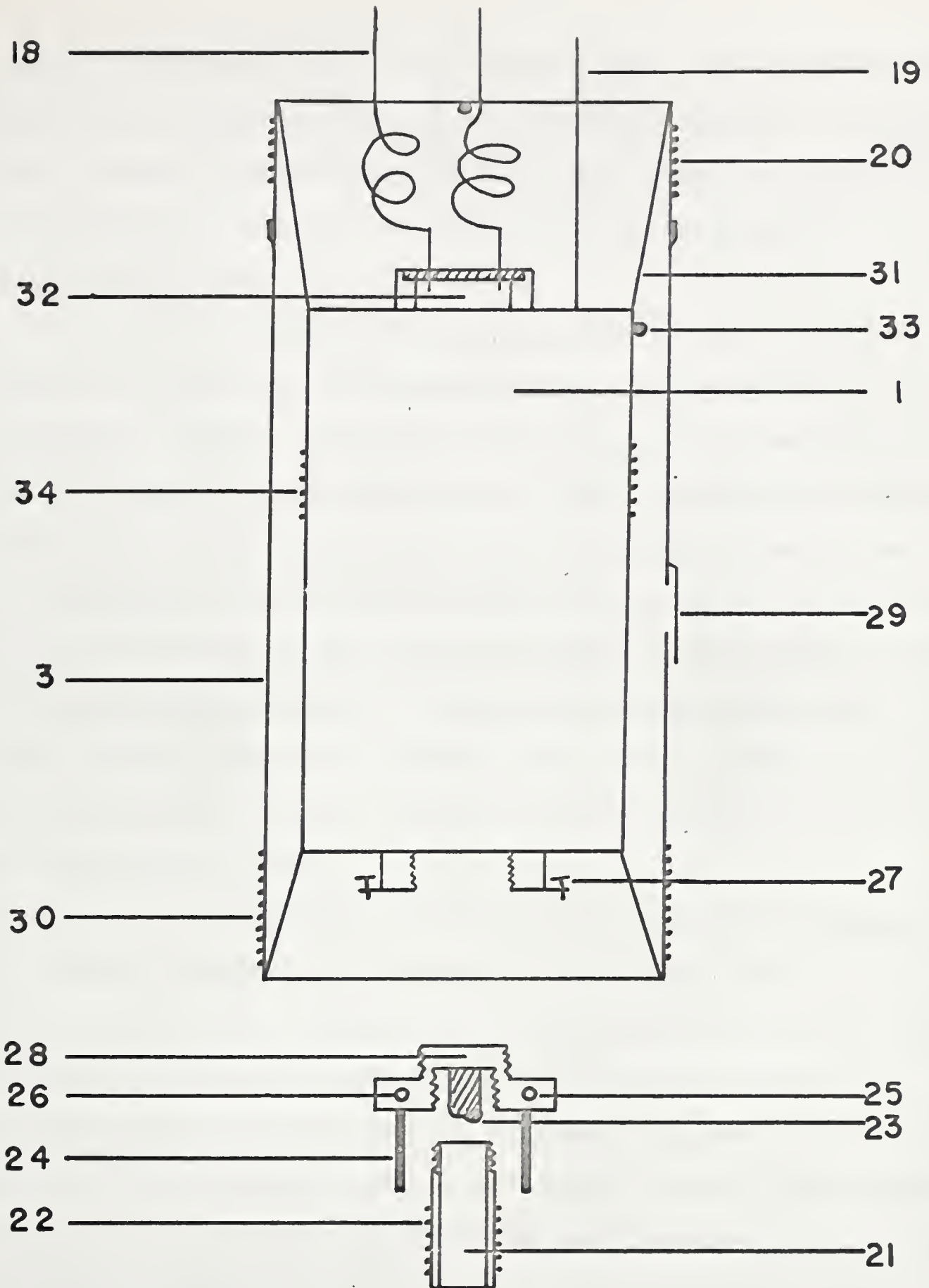


FIG. 3.1b

The gas thermometer bulb (1) is hung inside the adiabatic shield (3) by nylon threads (31) and the adiabatic shield is hung by nylon threads in a brass frame (2). The frame is screwed to a flat disc which, in turn, is thermally anchored to the bottom of the liquid helium container (7).

The cylindrical adiabatic shield is made of copper. The top of the adiabatic shield is detachable at the overlapping joint. There are three openings in the shield so that the space between the bulb and the shield is also evacuated. Copper sheets (29) placed over these openings prevent direct paths for radiation. On the outer surface of the shield is wound a 1000 ohms electric heater (20) of 40 SWG manganin wire. Plastiglo of Durathene Co. applied to the heater wires as they were wound served to cement them in place and to improve their thermal contact with the shield. There is a similar heater (34) on the gas thermometer bulb.

Adiabatic conditions are produced by controlling the shield temperature in response to a copper: gold + 2.1 at % cobalt differential thermocouple (33) which monitors the temperature difference between the shield and the gas thermometer bulb. One junction of the thermocouple is thermally anchored to the inside centre of the adiabatic shield and other junction near the top of the bulb with cigarette paper and nail varnish as insulator and cement respectively.

The experimental jacket (4) is a copper case surrounding the frame. It is attached by a wood's metal joint to the chrome plated copper vessel which is used as a helium reservoir of 1.5 litre capacity. Liquid helium is introduced into this vessel by means of syphon (10) which is an integral part of the cryostat. The evaporated helium gas is recovered through (11) via the heat exchanger (9). The space inside this jacket is evacuated by a thin walled one inch stainless steel tubing (5). Heat leak into the liquid helium is reduced by making all connections to it by stainless steel tubing. The central pumping line keeps the vacuum of 5×10^{-8} in the experimental chamber.

The Liquid helium can and experimental jacket are further surrounded by a radiation shield (8) cooled to liquid air temperature by thermally anchoring it to the liquid air container (13). The radiation shield is gold plated to reduce its emissivity. The assembly is enclosed in a vacuum tight jacket (15) of brass. The upper half of the jacket is fixed to a frame a sufficient distance from the floor to enable the removal of the lower section at the flanged joint with O-ring seal without having to lift the cryostat and without disconnection of electrical lines, and vacuum lines. This arrangement also provides a quick access to the experimental chamber without disturbing any other set up. This shield is chrome plated. The space inside this shield is evacuated by an independent vacuum system. The coupling of vacuum lines to the

vacuum systems is done via stainless steel bellows to eliminate strains.

Twenty-four electrical leads of 40 SWG silk covered copper wires enter the cryostat through a vacuum seal at room temperature made by pinching a polythene tube and filling with nail varnish. The leads reach down to the experimental chamber through the central pumping line (5). They are first anchored to liquid helium bath to reduce the heat leak along the leads. The heat leak could be further reduced by using finer wires, but as considerable manipulation of the lower part of the wire assembly at teflon terminals (27) was necessary, the use of sturdy leads was desirable. The electrical leads are further anchored to the adiabatic shield and gas thermometer bulb before going to the thermometers.

3.2 The Gas Thermometer

The bulb of the gas thermometer is made of copper. The wall thickness is $1/16$ " and volume is about 120 cc. It is drawn to scale in Fig. (3.1b). The stainless steel capillary of 0.3 mm bore (19) soldered to the top of the bulb provides a means for filling the bulb with helium and later on for the measurement of gas pressure in the constant volume gas thermometer bulb. The capillary is thermally anchored to the adiabatic shield, liquid helium and air stages to know the temperature distribution along the capillary length as is required for

dead volume correction. The other end of the capillary goes to the manometer described in next section. The length of capillary is kept as short as possible to reduce the dead volume.

3.3. Manometer System

Fig. (3.2) shows the mercury manometer designed for accurate measurement of gas pressure. The manometer arms are made of pyrex glass of 22 mm bore and 1.5 mm wall thickness. The short arm (5) is 13 cm long and the long arm 120 cm. The glass tubes are selected for straightness and uniformity of bore. The axis of the long arm is made vertical and in line with the axis of the short arm. The parallelism of the axes of the two arms of the manometer was achieved by clamping the tubes to two aluminum brackets which were fixed to the rigid frame and aligned to be vertical. The leveling of the manometer system is carried out by three leveling screws. Mercury heights are read on a meter bar (10) whose markings are in the plane of the axes of long and short arms of the manometer. Diffuse light with the background of slanting black and white lines is used for illuminating the mercury meniscus. The temperature of the mercury is noted by a mercury-in-glass thermometer dipping in a mercury bath adjacent to the manometer. The long arm was evacuated through a common vacuum line for the manometer system and the system for filling helium gas in the bulb. The gas filling system

FIGURE 3.2
DIAGRAM
OF THE
GAS THERMOMETER MANOMETER

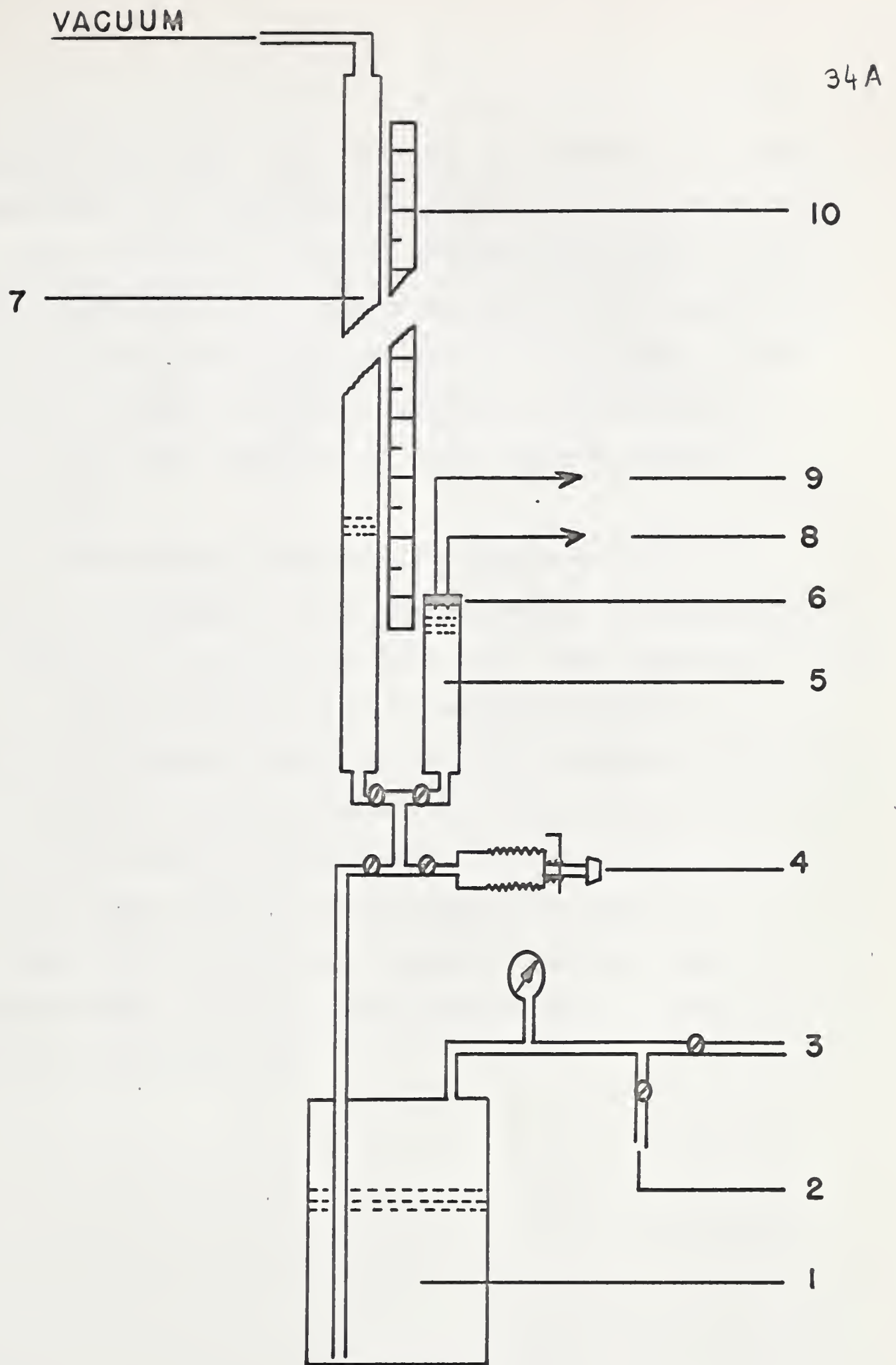


FIG. 3.2

will be described later. The short arm was closed with a stainless steel plug (6). This plug was vacuum sealed by De-Kotinsky cement. Two capillaries (8 and 9) soldered into the plug lead to the gas thermometer bulb and the filling system respectively. The ends of these capillaries were almost in the plane of the flat surface of plug so that the mercury in the short arm could be brought very close to the plug, thereby reducing the dead volume.

The mercury from the air-tight vessel (1) was forced into the manometer to the desired height by compressed air (2) and forced out by the vacuum line (3). Fine adjustment of the mercury height could be made by a mercury injector (4) which has a volume of about 10 cc. The manometer was used as a constant volume manometer by always setting the lower meniscus to very nearly the same mark. The readings of both menisci were taken relative to the nearest mm. mark on a standard meter bar in the plane of menisci using a Wild cathetometer KM268. Various corrections applied to pressure readings are mentioned later.

3.4 Gas Filling System

The system for filling the gas thermometer bulb is shown in Fig. (3.3). The capillary stopcock (9) is connected to the stainless steel capillary of 0.3mm bore, going to the plug cemented to the short arm of the manometer. Stopcock (12) provides connection to helium supply for filling the three litre reservoir (15) to about one atmosphere pressure and for flushing the bulb after it has been pumped for a couple of days. Vacuum line (14) goes to the long arm of the manometer with (13) going to the diffusion pump. Manometer (18) provides a rough estimate of cooling rate of the gas thermometer bulb when liquid air and helium are used for the purpose. When the bulb has been filled to the desired pressure, the stopcock (9) is closed. The dead volume of the constant volume gas thermometer is the volume of gas from this stopcock up to the point where the capillary enters the gas thermometer bulb.

The mercury manometer (17) is used to measure the vapour pressure of liquid helium contained in the vapour pressure chamber. Details of the manometer are given in the next section.



FIGURE 3.3
DIAGRAM
OF THE
HELIUM GAS FILLING SYSTEM

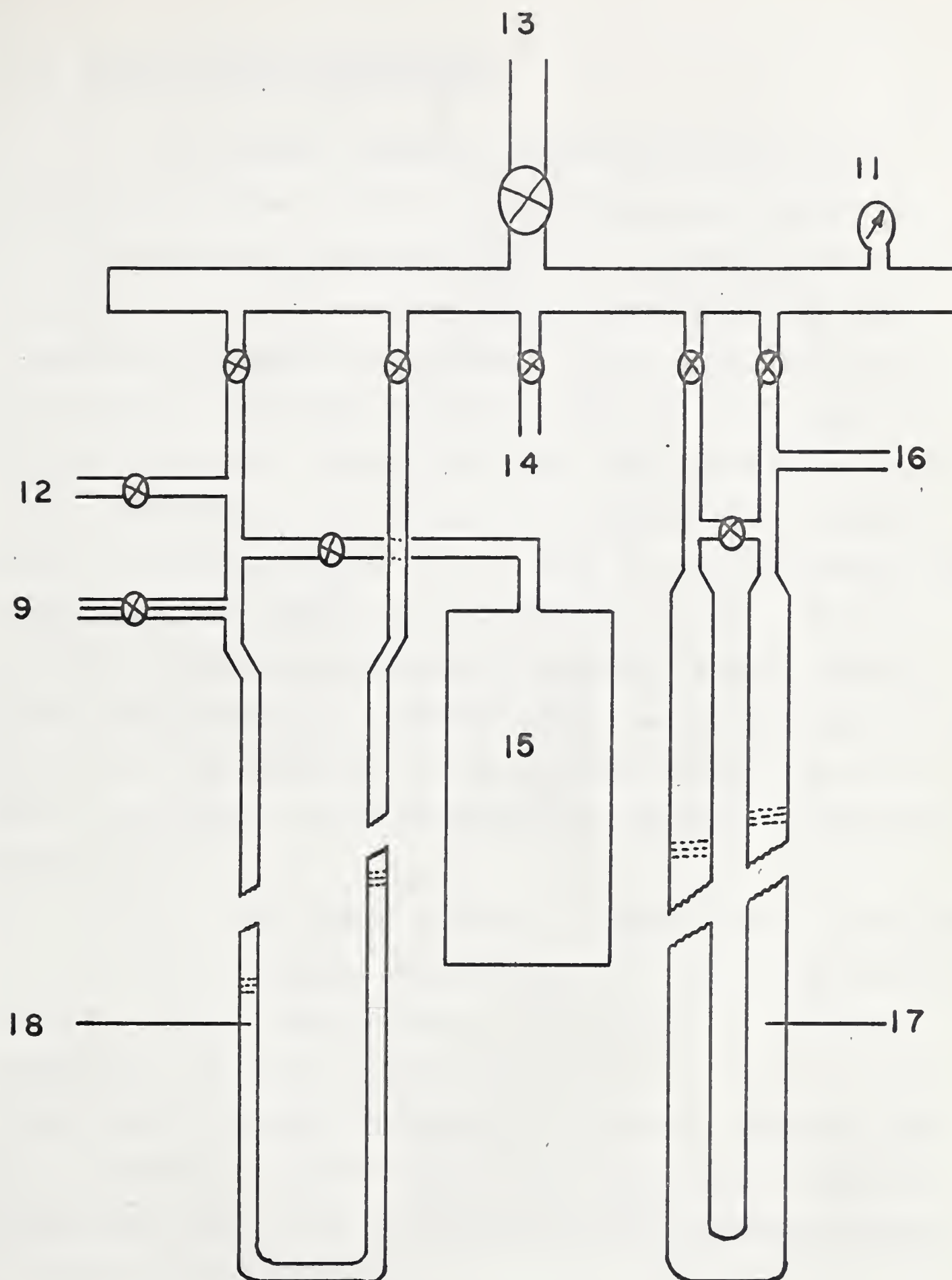


FIG. 3.3

3.5 Vapour Pressure Thermometer

The vapour pressure chamber (32) shown in Fig. (3.1b) has a volume of about 1.5 cc and was hard soldered on top of the gas thermometer bulb (1) to provide good thermal contact of the vapour pressure chamber with the object whose temperature is desired to be known. It was connected to two thin walled cupro-nickel capillaries (18) of 0.5 mm. bore in the lower part and 1.5 mm afterwards. Both capillaries were wound into spirals 25 cm long before making thermal contact with the adiabatic shield (3), in order to isolate thermally the vapour pressure chamber from the adiabatic shield. The capillaries were thermally anchored to adiabatic shield, liquid helium and air baths to minimize thermal conduction down the capillary. The dimensions of the pressure transmitting tubes were large enough to make thermomolecular pressure corrections negligible.

The vapour pressure of liquid helium was measured with a mercury manometer shown in Fig.(3.4). The manometer was of 19 mm bore to reduce meniscus corrections. The manometer temperature was known by a mercury-in-glass thermometer. In order that the mercury thermometer has the same response time as mercury in the manometer it was immersed in a mercury filled test tube of the same diameter as the manometer tubing to which it was fastened.

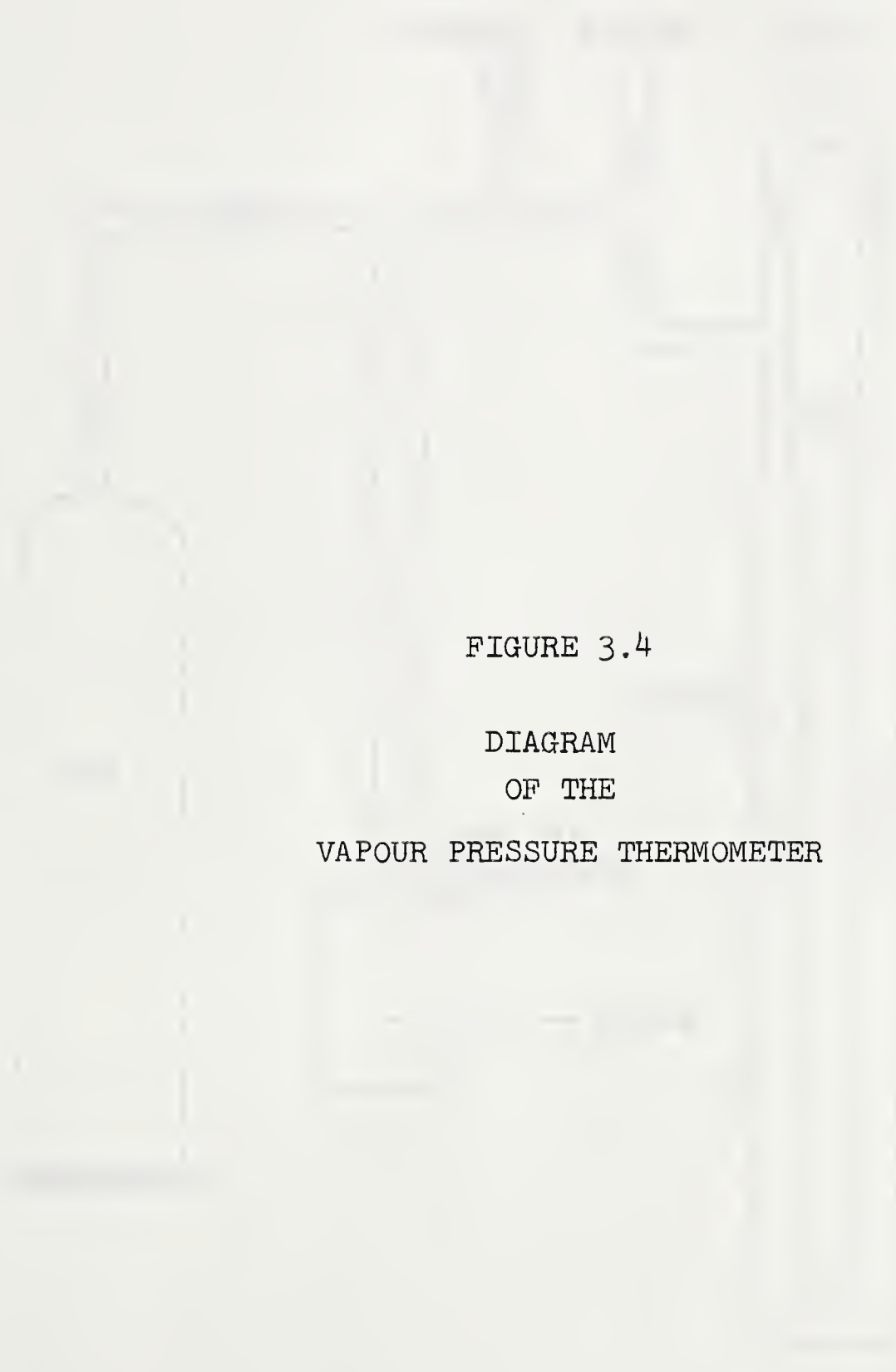


FIGURE 3.4

DIAGRAM
OF THE
VAPOUR PRESSURE THERMOMETER

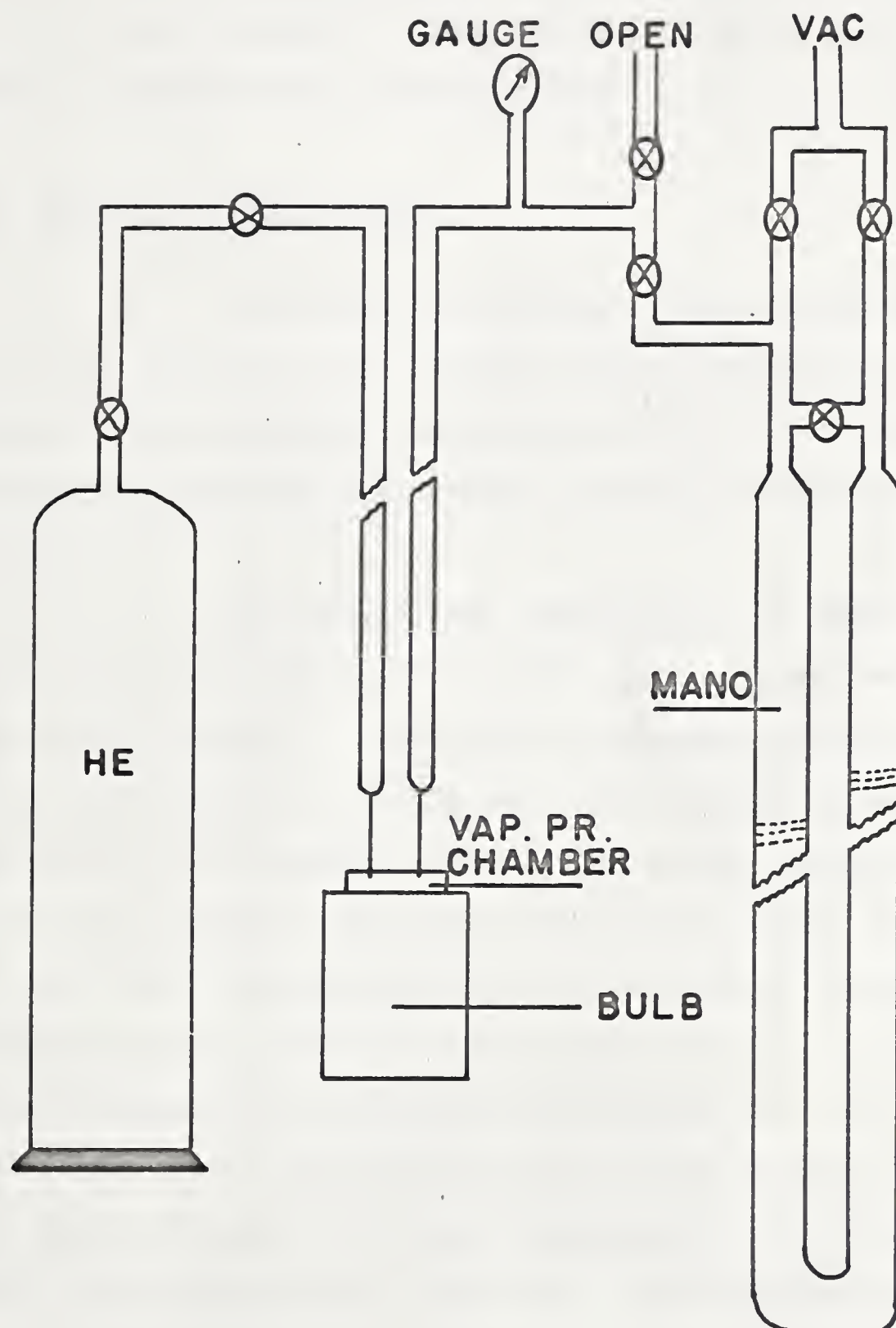


FIG. 3.4

The vapour pressure readings were subjected to gravity and density corrections and the 1958 ^4He scale of temperatures¹⁾ was used for finding the temperature corresponding to a given vapour pressure. The vapour pressure thermometer was used to calibrate the gas thermometer.

3.6 Resistance Thermometer

We calibrated two resistance thermometers, viz. a $\frac{1}{2}$ watt, 10 ohms, Allen Bradlêy carbon composition radio resistor and a germanium thermometer CG-1 from Radiation Research Laboratory, against the constant volume gas thermometer.

To reduce heat capacity and to improve thermal contact the insulating cover of the carbon thermometer was removed by grinding. Electrical insulation and good thermal contact were achieved by baking on a thin coat of G.9825 baking varnish of G.E. company. The varnish coated thermometer was placed into cavity (25), Fig. (3.1b), in a copper plug (28). The plug was later on screwed in the bottom of the gas thermometer bulb with silicone grease to ensure good thermal contact between the resistance thermometer and the bulb. The four leads of the thermometer were wrapped several times around the anchoring posts (24) and varnished down to minimize heat leak to the thermometer. The leads were thermally anchored to the

gas thermometer bulb, adiabatic shield and liquid helium stage to further reduce the heat leak.

The germanium thermometer cartridge was soldered to the copper plug mentioned above. The four leads were anchored in the same way as for carbon thermometer.

3.7 Resistance Measurement

The unknown resistance can be measured by comparing potential across a reference resistance with the potential across unknown resistance in series with it and connected across a battery. The comparison can be made indirectly by the use of some intermediate e.m.f., as may be supplied by the potentiometer. However, if the two e.m.f.'s are not constant then the above method is unsuited because the error introduced is essentially proportional to the length of time required to make measurement. The isolating potential comparator circuit (Fig. 3.5) of Dauphinee and Mooser¹⁴⁾ reduces the period of comparison to a fraction of a second.

By means of a double pole-double throw chopper M_3 the current flowing through reference resistance S and thermometer resistance X is reversed. Two choppers M_1 and M_2 are connected to S and X with condensers C_1 and C_2 bridging their moving contacts. Choppers M_1 and M_2 are driven synchronously with M_3 .

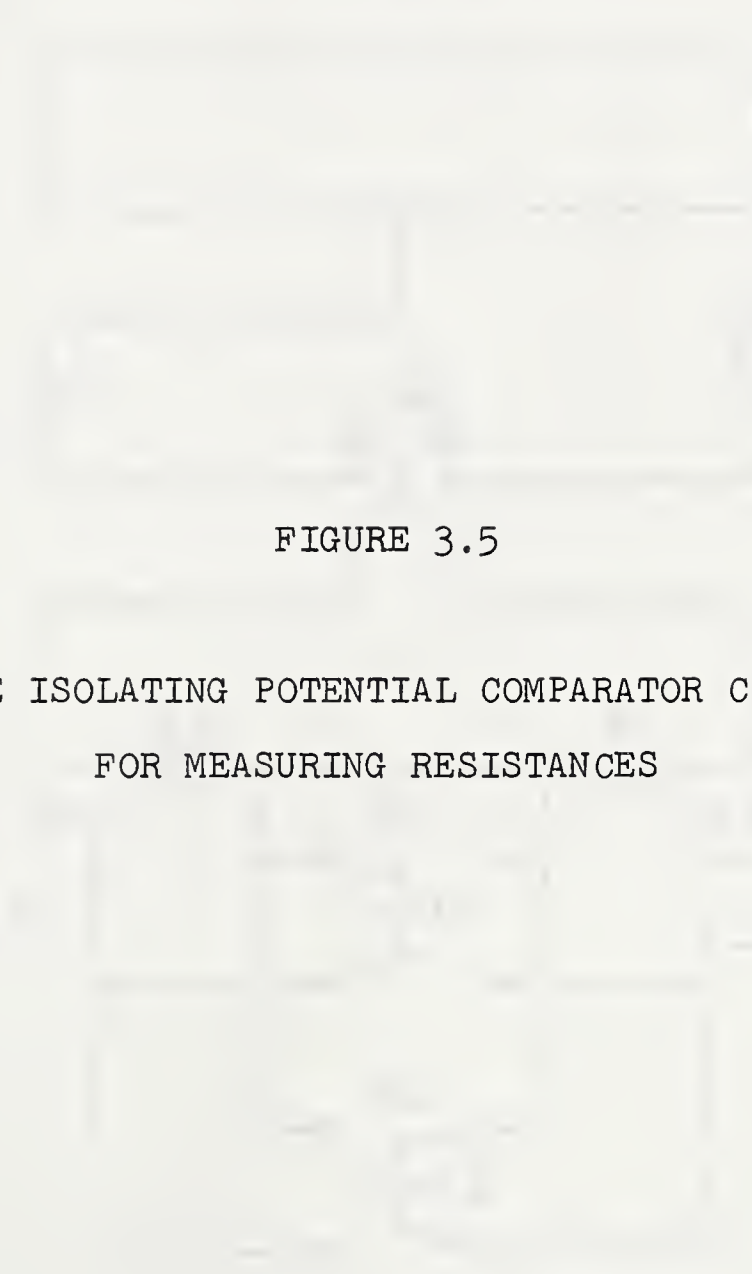


FIGURE 3.5

THE ISOLATING POTENTIAL COMPARATOR CIRCUIT
FOR MEASURING RESISTANCES

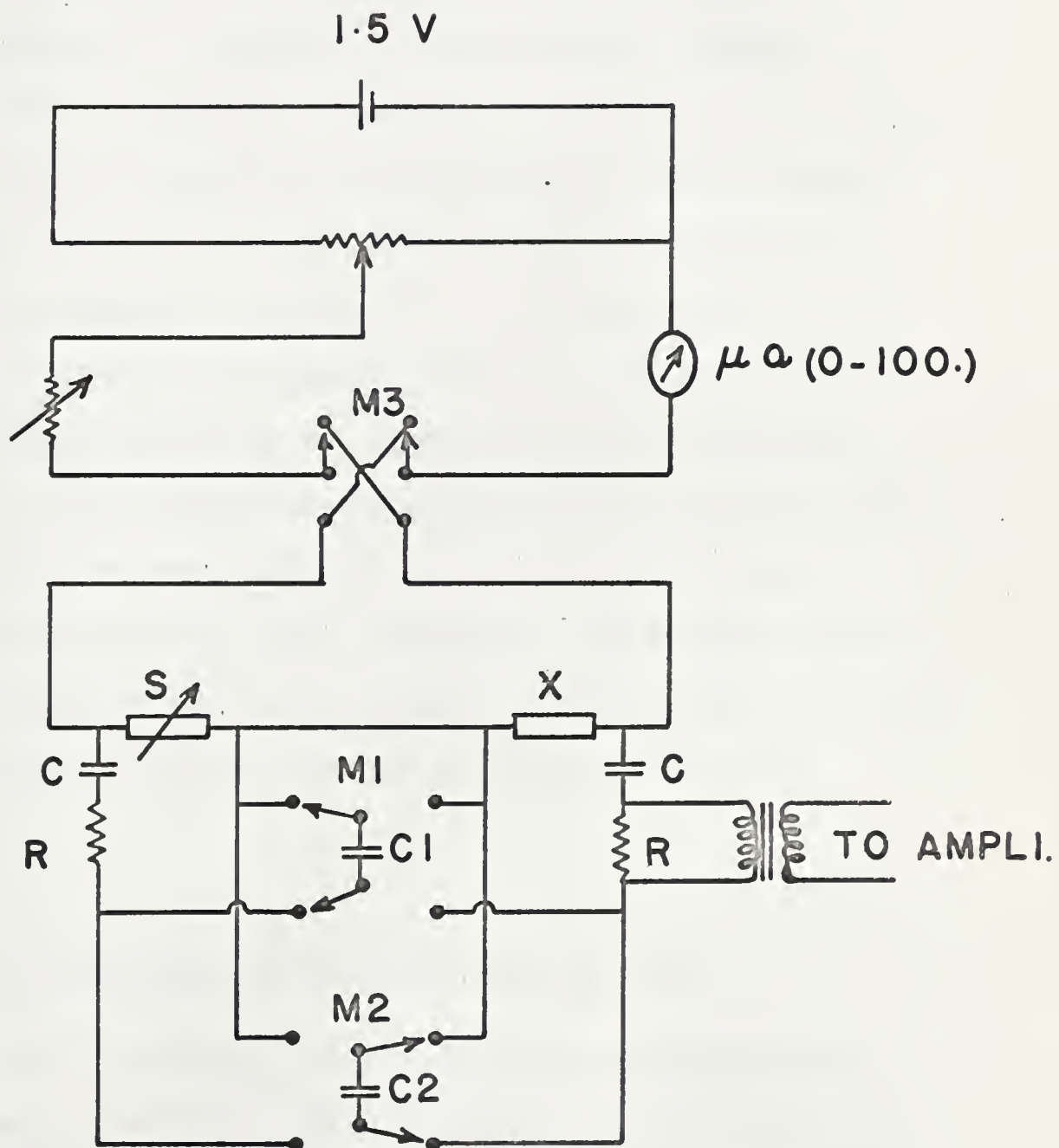


FIG. 3.5

With the arrangement shown in the figure C_1 is charged to V_s . Now the chopper operation transfers C_1 across X, meanwhile the polarity of battery is reversed. The condenser C_1 charges or discharges through R until it has the voltage V_x . As this process repeats a number of times per second, the result of any difference between V_x and V_s is a pulsating current through R. Similarly the operation of chopper M_2 causes a pulsating current through R which, in the chopper cycle, is spaced 180° apart from the one due to charging or discharging of C_1 through R. When $V_x = V_s$, the total e.m.f. around the loop is zero and no current flows through R.

The resistance of thermometers was measured by this circuit. The voltage across resistance R was magnified by electronic amplifier and applied to Leeds and Northrup Speedomax recorder used as a null detector. The resistance of the thermometer is given by the algebraic sum of decade resistors and the small differential resistance is deduced from the recorder reading.

3.8 Superconducting Transition Temperature of Lead

The transition temperature of 69 grade lead supplied by Consolidated Mining and Smelting Co. of Canada, was measured against the calibrated carbon resistance thermometer. The sample (23), Fig. (3.1b), was machined in the form of a

cylinder 1.45 cm long and 0.48 cm diameter. It was soldered by bismuth-cadmium solder to the copper plug (28) screwed to the bottom of bulb (1). The transition temperature was determined by mutual inductance bridge of Cryotronics Inc. The primary coil (30) of 300 turns was wound on the adiabatic shield (3) and the secondary coil (22) of 200 turns on the perspex former (21) surrounding the lead sample. The null balance of the bridge was observed on the oscilloscope.

CHAPTER IV

EXPERIMENTAL PROCEDURE

The gas thermometer bulb, the vapour pressure chamber, the inner and outer jackets are evacuated to a pressure of 2×10^{-6} or better. The bulb is flushed with helium gas and is connected to a helium reservoir for filling. Helium gas is circulated through the liquid helium container and then the free end of the built-in syphon is closed to avoid condensation of moisture in the system.. Liquid air container is now topped. After the radiation shield has come to the temperature of liquid air, exchange gas is admitted into the experimental chamber. Initial cooling of the bulb is done by liquid air in the helium can and by pumping on the liquid air further cooling down to about 65°K is done. Liquid air is then forced out by a small pressure of helium gas. At this point exchange gas is pumped out of the inner system and liquid helium is now transferred to the helium can at a slow rate. Further cooling of the bulb is accomplished by circulating cold helium through the vapour pressure chamber. As the cooling proceeds liquid helium condensing in the tube drips down in the vapour pressure chamber where it re-evaporates, absorbing heat. Refluxing continues until the temperature of the bulb is sufficiently low for liquid to collect in the vapour pressure chamber. This procedure while giving the

desired end result was found to be quite time consuming, since the heat of vaporization of liquid helium is low and the passage of liquid down the tube is inhibited by the back current of gas from the evaporating helium. To effect a considerable saving of cooling time we made use of exchange gas.

About 8 liters of liquid helium are used to initially cool the cryostat and fill the 1.5 litre can. About $2\frac{1}{2}$ hours are required for cooling operation to liquid helium temperature after the initial cooling to liquid air temperature has been done. Liquid air in the 2.5 litre can lasts for about 8 hours and liquid helium in the helium can for 12 hours. The sources of heat inflow are (1) conduction through low-pressure gas in vacuum chambers (2) radiation (3) conduction of heat along tubes or electrical leads (4) Joule heating. These factors were taken into account in the design of cryostat.

After the gas thermometer bulb has been cooled to liquid helium temperature and it has helium gas at the desired pressure, it is disconnected from the filling system at the capillary stopcock to serve as a constant volume gas thermometer. Helium is now condensed in the vapour pressure chamber and after the bulb has acquired a constant temperature the exchange gas is pumped out. When vacuum of about 10^{-7} is obtained, the vapour pressure on the liquid helium in the vapour pressure chamber is reduced slightly whereupon the temperature adjusts itself accordingly. When the bulb has acquired a constant

temperature, the vapour pressure of liquid helium, the gas thermometer manometer and the resistances of carbon and semiconductor thermometers are noted simultaneously. This provides one calibration point. By further pumping on the liquid helium more calibration points are obtained using the 1958 ^4He Scale of Temperatures. The liquid in the vapour pressure chamber is now removed by heating and pumping. From now on the measurements proceed.

The bulb is heated until the thermometer reaches a desired value and the bulb heater is switched off. The heater power to adiabatic shield is regulated manually to control the shield temperature. The temperature difference between bulb and shield is indicated by a differential thermocouple and noted on a galvanometer. Fine adjustment of the shield heater power is carried out with the help of a Leeds and Northrup Speedomax chart recorder. This is done by watching the trace on the chart and observing whether the resistance thermometer is cooling or warming, meaning thereby that the shield is cooler than bulb or vice versa. The adjustment of shield heater power is carried out accordingly until the resistance thermometer shows negligible temperature drift. At each steady temperature, three

observations are taken at about three minute intervals to ascertain that the temperature of the bulb is constant, and that the pressure system is at equilibrium, as evidenced by constant pressure readings on the manometer within the experimental error of 0.002 cm.

4.1 Conversion of Vapour Pressure to Temperature

The vapour pressure was measured against a glass mirror scale in millimeters mercury at room temperature and local gravity of 981.155 cm/sec^2 . This pressure was corrected to mm.Hg at 0°C and reduced to standard gravity using $h_0 = hg/980.665$, where g is local gravity and h is the pressure after density correction. The temperature was obtained from the 1958 ^4He scale of temperatures ¹⁾.

4.2 Conversion of Gas Thermometer Pressure to Temperature

Temperatures¹⁵⁾ were calculated from the relation

$$(4.1) \quad \dots \quad P \frac{v_b}{T(1+\frac{B}{V})} + P \sum_{i=1}^6 \frac{v_i}{T_i(1+\frac{B_i}{V_i})} = \text{constant},$$

where P is the pressure of gas,

v_b is the volume of gas thermometer bulb,

B is the second virial coefficient,

V is the molar volume,

v_i is the part of the dead volume at temperature T_i .

The dead volume is divided into six sections $v_1, v_2, \dots, v_6$, corresponding to regions of constant temperature or to regions for which the thermal gradient is known. The temperatures (or effective temperatures for regions of thermal gradient) corresponding to these volumes are indicated by $T_1, T_2, \dots, T_6$. The temperature distribution along the capillary is shown in Fig. (4.1). The effective temperature of gradient was determined by integrating the following expression:

$$(4.2) \quad \dots \quad \frac{v_i}{T_i(\text{eff})} = \int \frac{A d\ell}{T}.$$

where A is cross-sectional area of capillary and $d\ell$ is differential length. On the assumption of linear temperature distribution along the portion of capillary of length L with

FIGURE 4.1

TEMPERATURE DISTRIBUTION ALONG THE
GAS THERMOMETER CAPILLARY

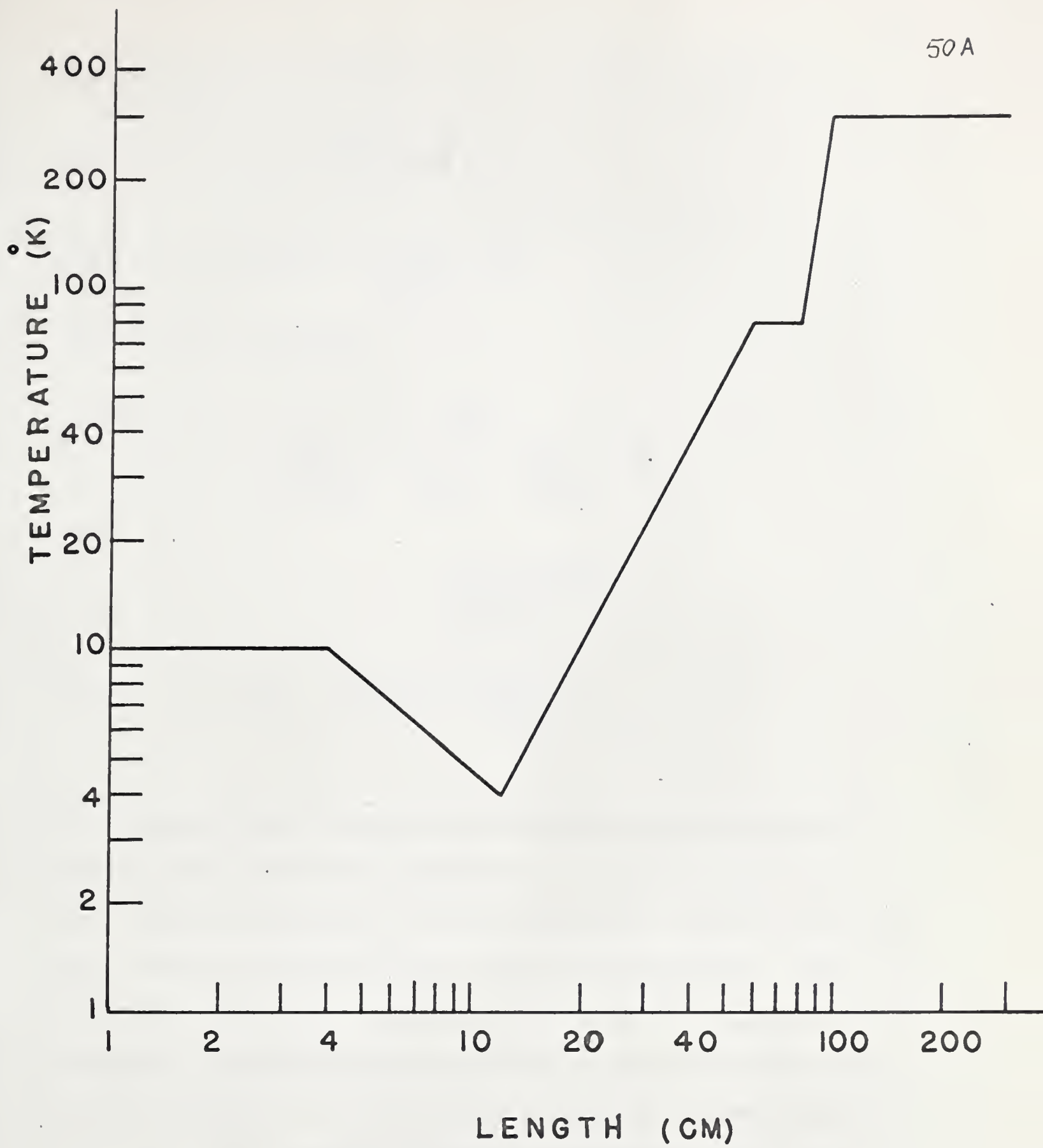


FIG (4.1)

the end points at temperatures T_1 and T_2 , we can write:

$$T = \frac{T_2 - T_1}{L} \ell + T_1$$

$$(4.3) \dots \text{and } d\ell = \frac{L}{T_2 - T_1} dT$$

Using (4.3) in (4.2)

$$\begin{aligned} \frac{v_i}{T_i(\text{eff})} &= \int_{T_1}^{T_2} \frac{AL}{T_2 - T_1} \frac{dT}{T} \\ &= \frac{v_i \ln T_2/T_1}{T_2 - T_1} \end{aligned}$$

$$(4.4) \dots \text{or } T_i(\text{eff}) = \frac{T_2 - T_1}{\ln \frac{T_2}{T_1}}$$

This method of calculating the effective temperature gave results which differed significantly from the arithmetic mean only for gradients between liquid air and helium baths and between liquid air bath and room temperature. The assumption of linear temperature distribution along the capillary is sufficiently accurate as the dead volume correction is small in view of the large bulb volume used.

The constant on right hand side of eq. (4.1) depends on the amount of gas in the system and is determined by calibrating at known temperatures. This calibration was

obtained near the normal boiling point of helium from the 1958 temperature scale for ^4He .

The molar volume V was calculated from the equation of state,

$$(4.5) \quad PV = RT\left(1 + \frac{B}{V}\right).$$

where R is gas constant. Solving this for V and expanding to second order, we get:

$$(4.6) \quad \bar{V} = \frac{RT}{P} \left[1 + \frac{BP}{RT} - \left(\frac{BP}{RT}\right)^2 \right]$$

Eq. (4.6) can be re-written (to second order):

$$\frac{TB}{V} = \frac{BP}{R} \left[1 - \frac{BP}{RT} + 2 \left(\frac{BP}{RT}\right)^2 \right]$$

and is substituted for the term containing the molar volume in Eq. (4.1). For the filling pressures used the higher terms are negligible. We have used the adopted values of second virial coefficient for helium by Keesom¹⁹⁾ because they are in much better agreement with the experimental determinations than the calculated values of Kilpatrick and others.²³⁾ The corrections due to third virial coefficient are negligible. Non-ideality correction for the gas in dead volume, which is represented by the term $T \frac{B_1}{V_1}$ in Eq. (4.1), was found to be negligible.

4.3 Corrections in Constant Volume Gas Thermometer

1. Dead volume correction:

This correction was calculated from the known dimensions of capillary and manometer. The covolume of meniscus in the short arm of the manometer was obtained from the empirical formula¹⁶⁾ by Kistemaker. The author has also drawn up a table for the volumes of menisci in terms of tube radius and meniscus height which is convenient for dead volume correction.

2. Correction for thermal expansion of bulb:

This was based on the linear thermal expansion data¹⁷⁾ of Beenakker and Swenson. The volume expansion was derived on the assumption of isotropy of bulb material. The volume of gas thermometer bulb remains constant from 4 to 20°K.

3. Correction due to thermomolecular pressure difference in the capillary:

This correction arises when the mean free path of gas molecules is comparable with the width of the capillary connecting the thermometer bulb to the manometer.

The tables¹⁸⁾ given by Roberts and Sydoriak were used.

4. Correction for non-ideality of gas:

The virial coefficients used were taken from Keesom's book¹⁹⁾. The third virial coefficient is very small and the terms containing it were neglected. The non-ideality of helium produces a significant correction, particularly at high pressures and low temperatures. Thus to minimize the non-ideality corrections the gas pressures used were kept low.

5. Manometry Corrections:

- a) Capillary depression in both arms of the manometer: The table²⁰⁾ given by Kistemaker was used for this correction.
- b) Meter scale correction for ruling error and extension of scale under its own weight: The correction for the extension of meter bar was not applied since the scale was equally supported at the upper and lower ends. The ruling error checked by cathetometer was negligible.

c) Correction for temperature of mercury other than 0°C and for acceleration due to gravity other than 980.665 cm/sec^2 was made.

6. Loss of gas:

This was taken care of by finding the pressure of gas when the apparatus was brought back to room temperature after cooling to liquid helium temperature. The pressure of gas was found to be same after cycling.

7. Correction for expansion of bulb with pressure was not considered as the bulb wall is $1/16$ inch thick and the maximum gas pressure was below one atmosphere.

4.4. Resistance Thermometers

Resistance thermometers were calibrated against the gas thermometer. In the resistance measurement of semiconductor thermometer a measuring current of 10 micro-amperes was used. This current could be increased considerably without materially altering the resistance at a given temperature, indicating reasonable insensitivity to measuring current. Of course, the effect of measuring power on thermometer resistance depends on thermal contact of the sensing element with heat sink. A poor thermal contact of element with bath

causes greater power dependence. To achieve good thermal contact we had soldered the thermometer cartridge with the heat sink. By filling the cartridge with helium gas, response of this thermometer could have been enhanced by decreasing the thermal relaxation time between the sensing element and cartridge (heat sink) which was at the temperature of the gas thermometer bulb. This was, however, not done due to the possibility of change in characteristics of thermometer by adsorbed helium. To provide good thermal contact to the heat sink the plastic cover of the carbon thermometer was ground off. The electrical leads were thermally anchored to the heat sink to reduce heat leak to the thermometers..

The superconducting transition temperature of lead was obtained by making the resistance measurements of calibrated carbon thermometer in the vicinity of transition temperature and simultaneously measuring the mutual inductance. Plotting the temperature versus mutual inductance, the transition temperature was taken as mid point of the curve on the inductance axis. Carbon thermometer was calibrated by gas thermometer at two different filling pressures. The transition temperature was obtained at these filling pressures of gas thermometer and their mean value was taken to be the transition temperature.

CHAPTER V

RESULTS AND DISCUSSION5.1 Carbon Thermometer

Fig. (5.1) shows the calibration curve of 10 ohms $\frac{1}{2}$ W, Allen Bradley carbon thermometer. For interpolation of temperature the two constant formula of Clement and Quinell was used,

$$(2.24) \quad \left(\frac{\log R}{T_c} \right)^{\frac{1}{2}} = a + b \log R$$

where a and b are constants to be determined experimentally and R is resistance in ohms. The constants evaluated at 4.144°K and 19.882°K are $a = -0.68688$ and $b = 0.71981$. From the above equation the interpolated temperature T_c corresponding to a given R was calculated. The deviation of interpolated temperature from the gas thermometer temperature has been plotted against the true temperature in Fig. (5.2). Temperatures calculated from resistance values using this two-constant equation are within ± 0.7 percent of the measured temperatures from 4° to 20° Kelvin. Clement and Quinell ⁶⁾ have reported the calculated temperatures to be within $\pm 0.5\%$ using the three-constant formula (2.22).

The sensitivity of carbon thermometer increases with decrease of temperature as shown in Fig. (5.2A). The

FIGURE 5.1
CALIBRATION CURVE
FOR THE
CARBON THERMOMETER

CARBON THERMOMETER.

58 A

RUN B1 x

RUN B2 o

RUN B3 .

RESIS.
OHMS

TEMPERATURE (K) →

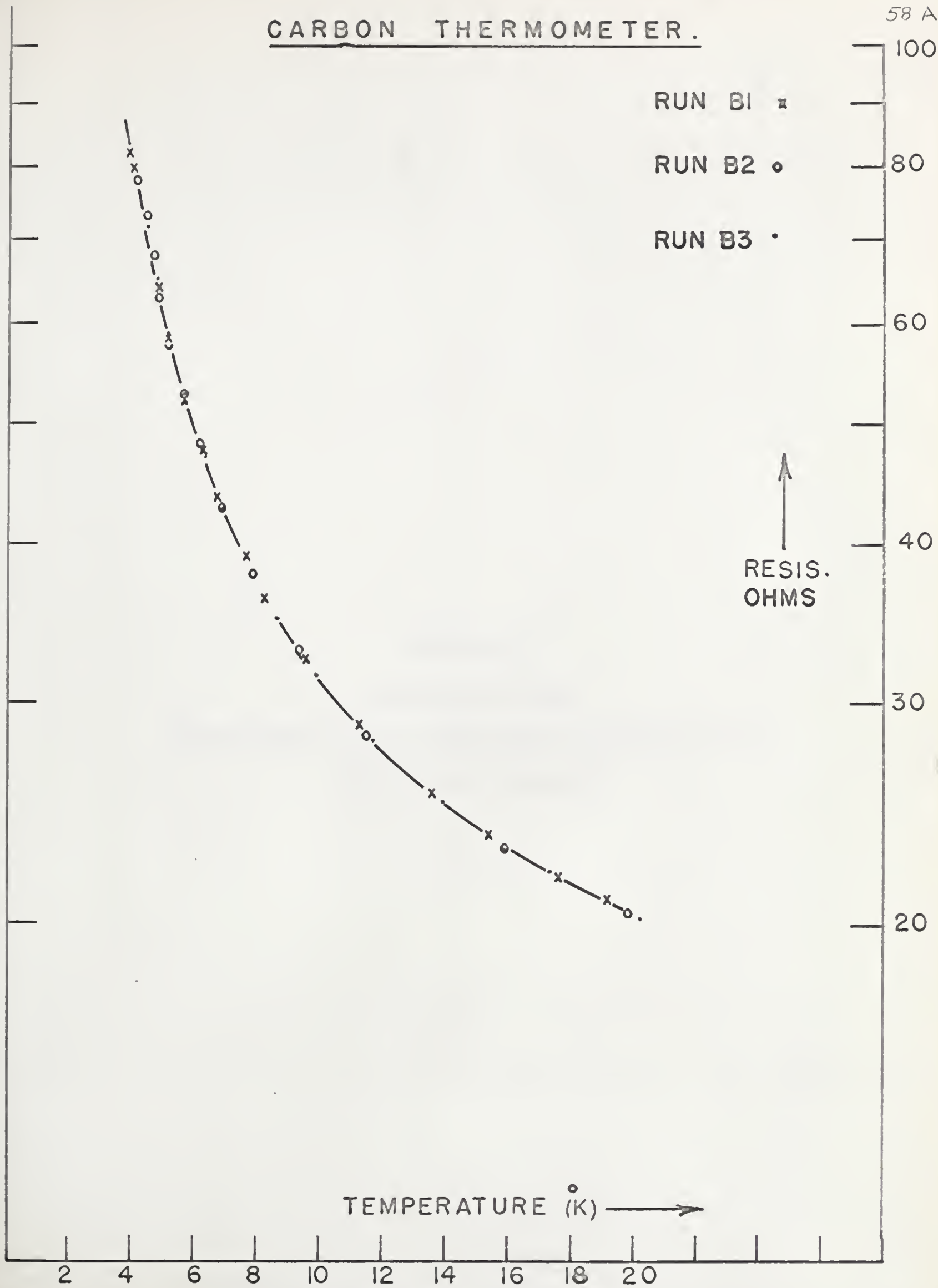




FIGURE 5.2

DEVIATION OF THE

INTERPOLATED CARBON THERMOMETER TEMPERATURE SCALE

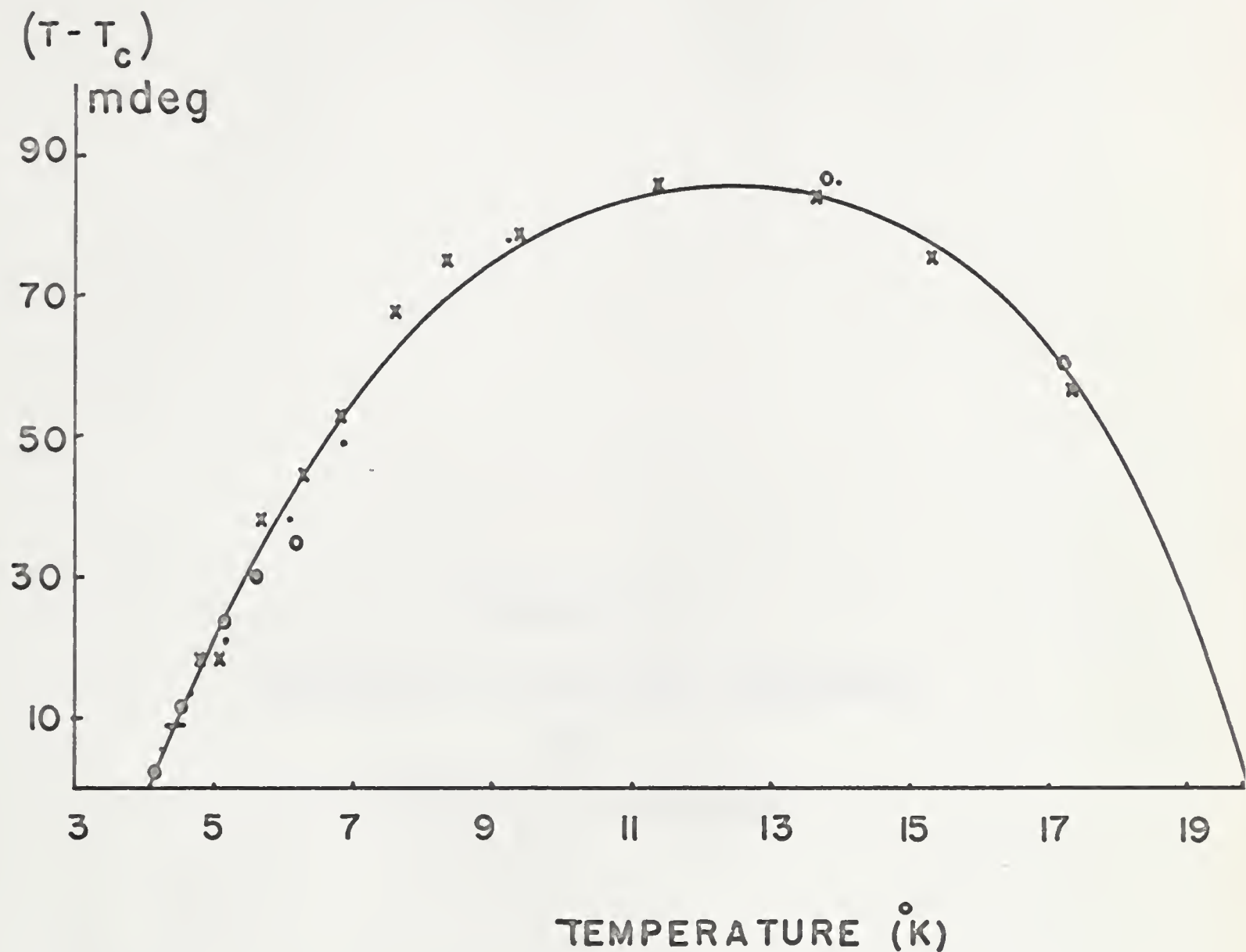
FROM TRUE TEMPERATURE

RUN B1 x

RUN B2 o

RUN B3 .

59 A



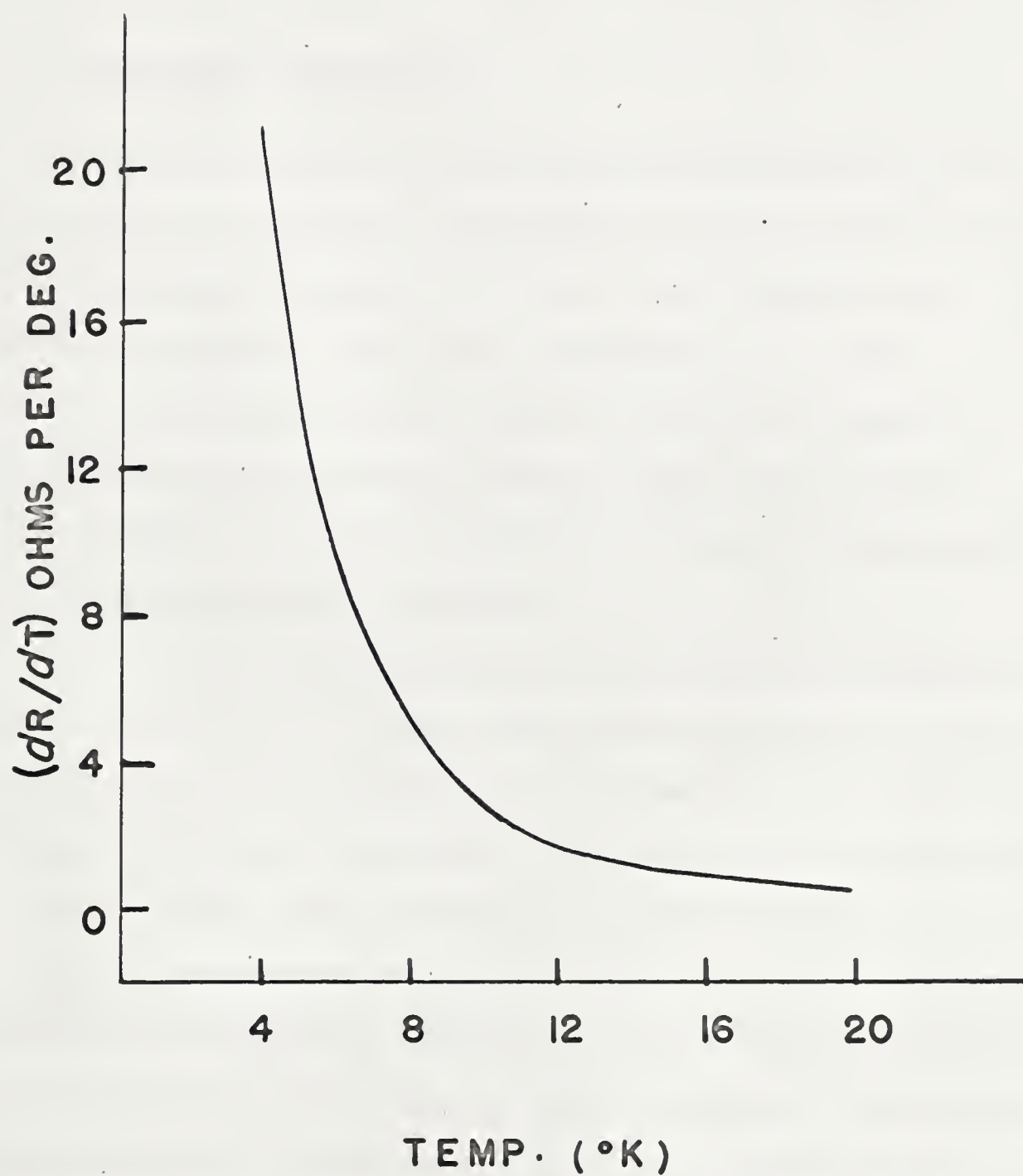
CARBON RESISTANCE THERM.

ALLEN BRADLEY (10 OHMS)

FIG. 5.2

FIGURE 5.2 A
SENSITIVITY OF THE CARBON THERMOMETER
AS A
FUNCTION OF TEMPERATURE





SENSITIVITY OF CARBON THERMOMETER.

FIG. 5.2A

reproducibility at liquid helium temperature is ± 3 mdeg on thermal cycling between helium temperature and room temperature. This is comparable to the findings of Clement and Quinnet on the Allen Bradley Carbon thermometers.

5.2 Germanium Thermometer

The resistance of germanium thermometer CG-1 as a function of reciprocal temperature is shown in Fig. (5.3). The activation energy E_3 (cf. Eq. 2.28) measured from the slope of graph at the lowest temperature is 3.941×10^{-4} ev. The conductivity in this range is due to the presence of compensating acceptors which produce empty donor states. Such a curve has been observed in lightly compensated²¹⁾ n-or p-type germanium by Fritzsche.

The calibration curve of germanium thermometer is shown in Fig. (5.4). This characteristic serves to show the suitability of a material for thermometry. We notice that the transition between extrinsic conduction and impurity conduction is near 11.0°K . The transition is quite abrupt and there is a bump in characteristic at the place where the two hyperbolic components intersect. Blakemore²²⁾ has observed similar characteristics of a doped germanium with 8% compensation with antimony. Sensitivity, $\frac{dR}{dT}$, of CG-1 as a function of temperature is shown in Fig. (5.4A). The minimum in sensitivity curve lies at the temperature where point of inflection occurs.

FIGURE 5.3

THE VARIATION OF RESISTANCE WITH THE
RECIPROCAL OF THE ABSOLUTE TEMPERATURE
FOR THE GERMANIUM THERMOMETER

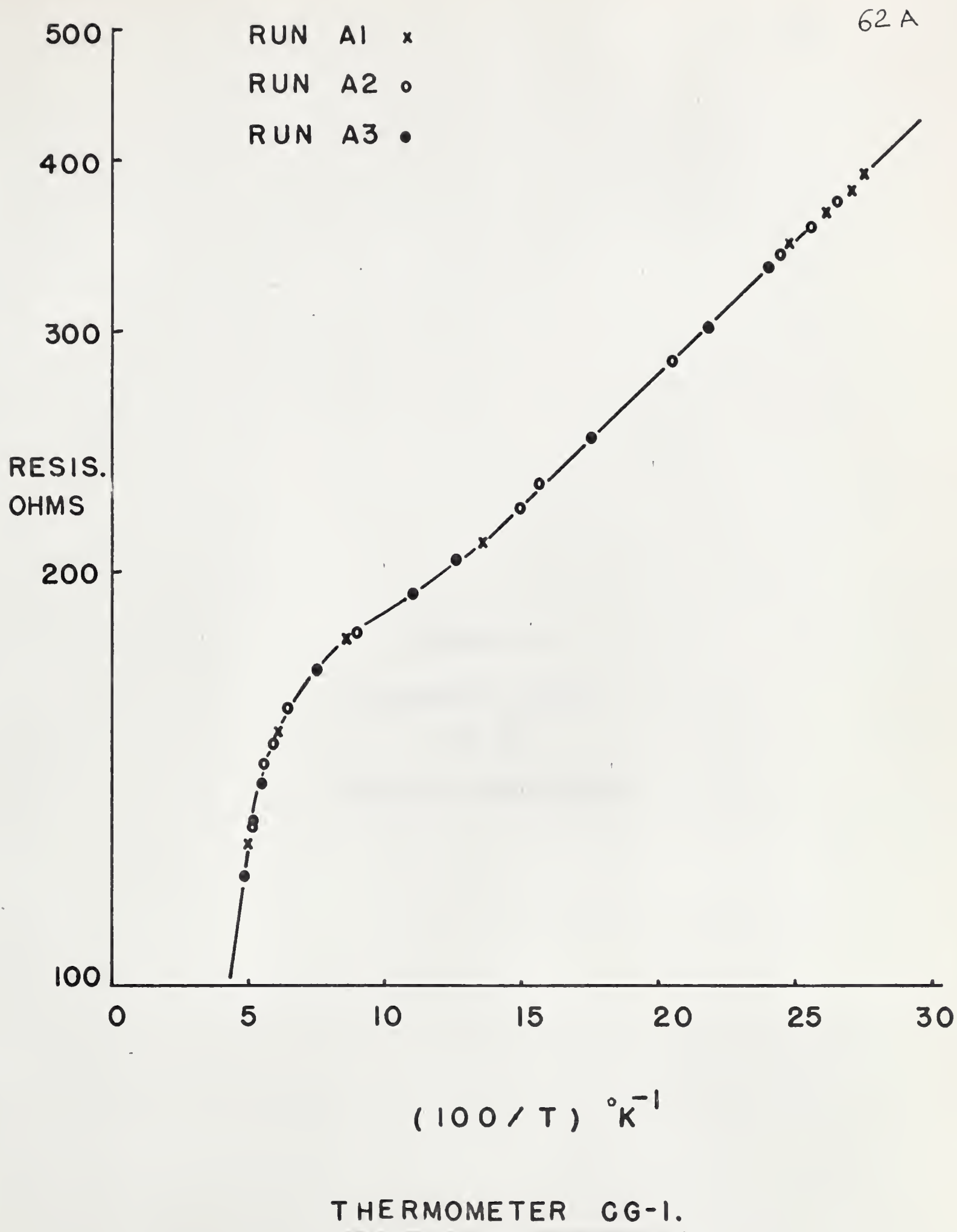
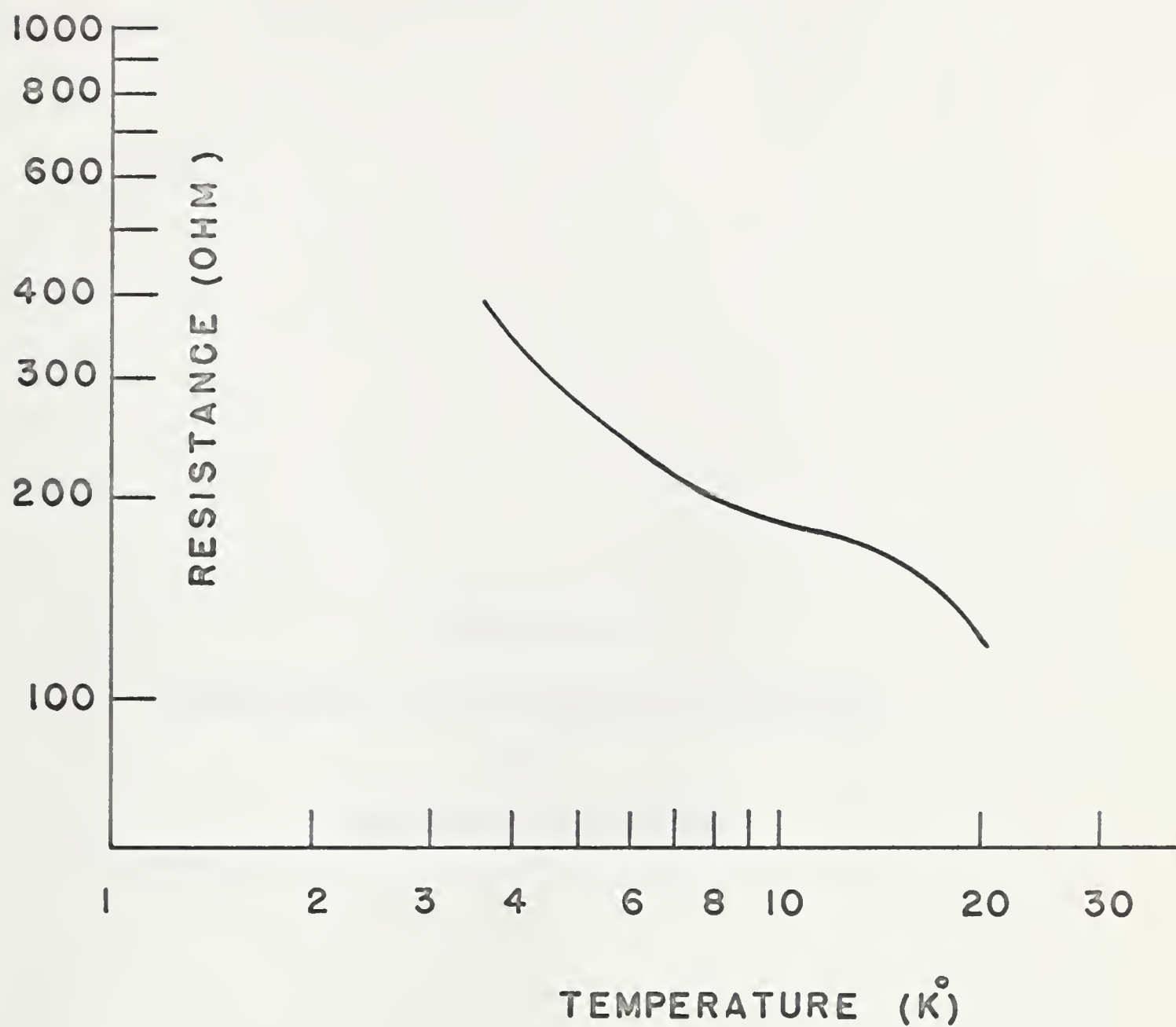


FIG. (5.3)



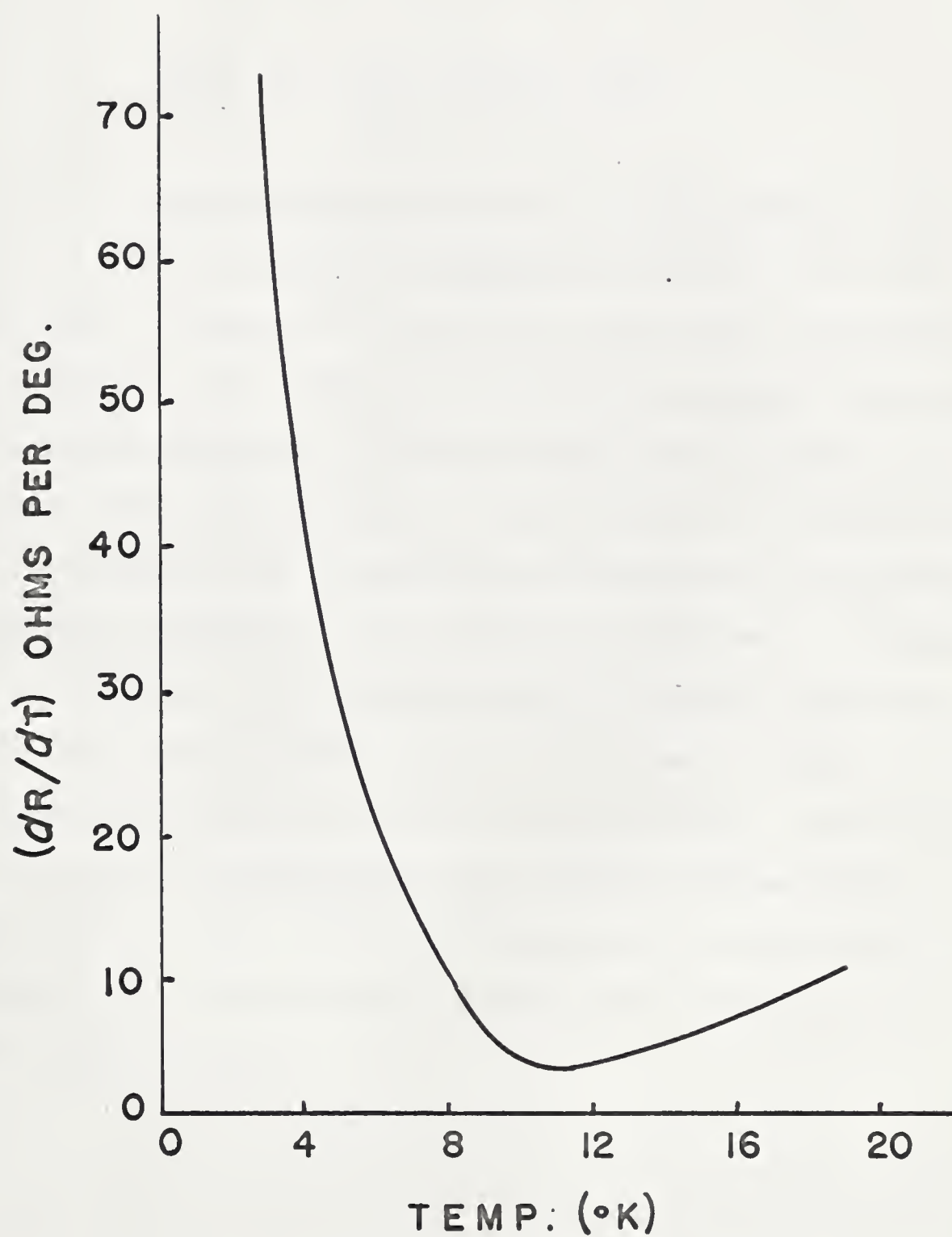
FIGURE 5.4
CALIBRATION CURVE
FOR THE
GERMANIUM THERMOMETER



GERMANIUM THERMOMETER
(CG-1)

FIG. 5.4

FIGURE 5.4 A
SENSITIVITY OF THE GERMANIUM THERMOMETER
AS A
FUNCTION OF TEMPERATURE



SENSITIVITY OF GERMANIUM THERM.

FIG. 5.4A

The empirical formula used for finding the interpolated temperature T_c corresponding to a given R is:

$$(5.1) \quad \log_{10} R = \sum_{n=0}^7 a_n (\log_{10} T_c)^n$$

Temperatures calculated by this equation are within $\pm 0.4\%$ of measured temperatures from 3° to 20°K , except near the region of inflection where the interpolated temperatures T_c lie within $\pm 1.1\%$ of the measured temperatures. The coefficients a_0 to a_7 were evaluated with the help of I.B.M. Computer 7040 by the method of least squares. It was found that the deviation of interpolated temperature from measured temperature decreased as the number of terms was increased in the polynomial. The coefficients of Seventh order polynomial are given in Table 5.2.1. Run A_1 was a trial run and the results of this run are not included here. The deviation of interpolated temperatures from measured temperatures is shown in Fig. (5.5). It is seen that the interpolation formula is not satisfactory in the neighborhood of bump in R-T characteristic.

TABLE 5.2.1

Coefficients	Run A_2	Run A_3	Run A_2/A_3
a_0	2.60827	2.53581	5.35648
a_1	15.04952	0.53608	-12.84764
a_2	-90.61040	-17.68076	23.80397
a_3	230.98796	52.59533	-17.77599
a_4	-320.22113	-78.94948	-8.14611
a_5	250.43849	65.71119	23.29163
a_6	-103.47280	-28.33764	-14.27061
a_7	17.48890	4.85660	2.85787

FIGURE 5.5

DEVIATION OF THE INTERPOLATED GERMANIUM THERMOMETER
TEMPERATURE SCALE FROM TRUE TEMPERATURE

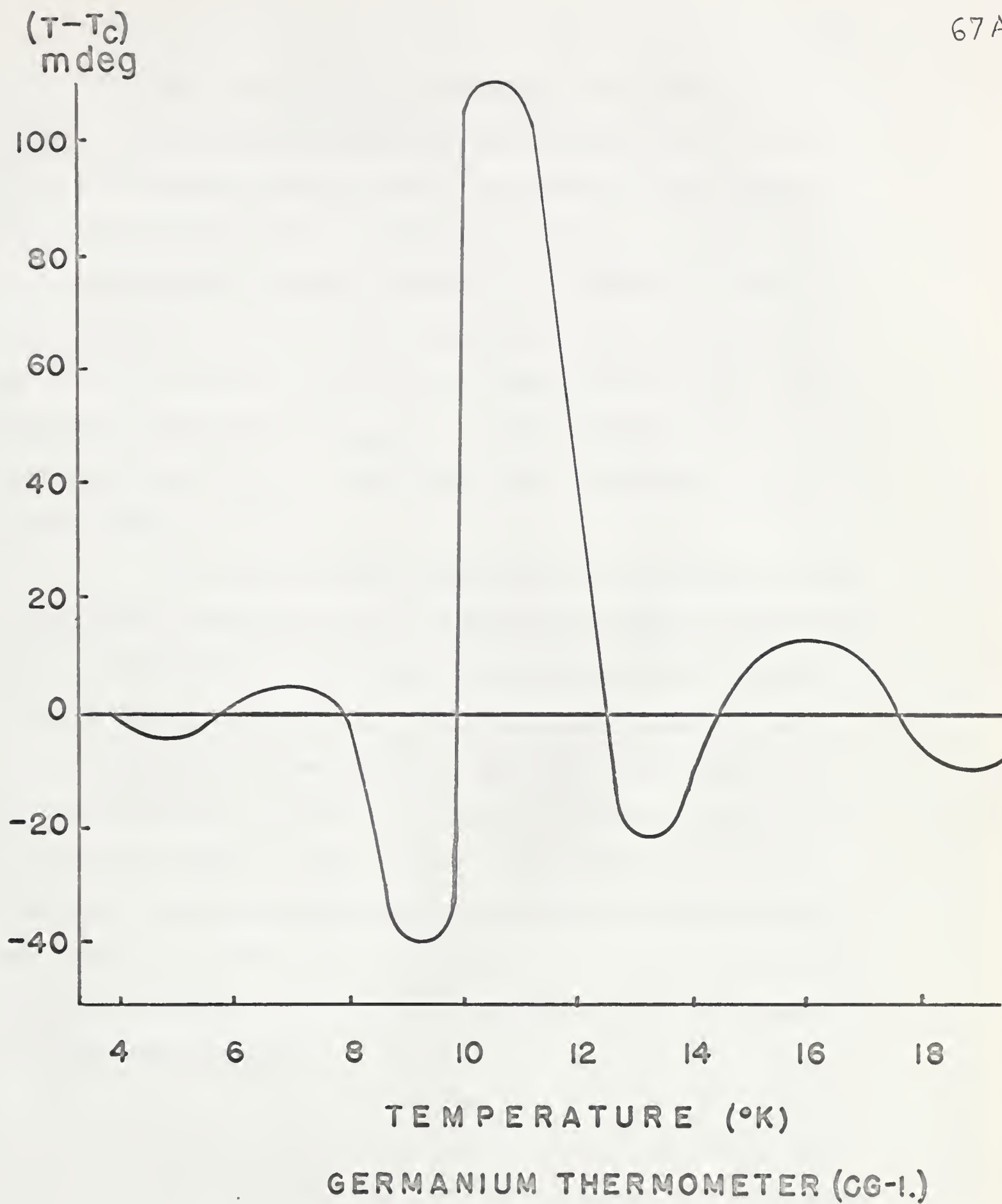


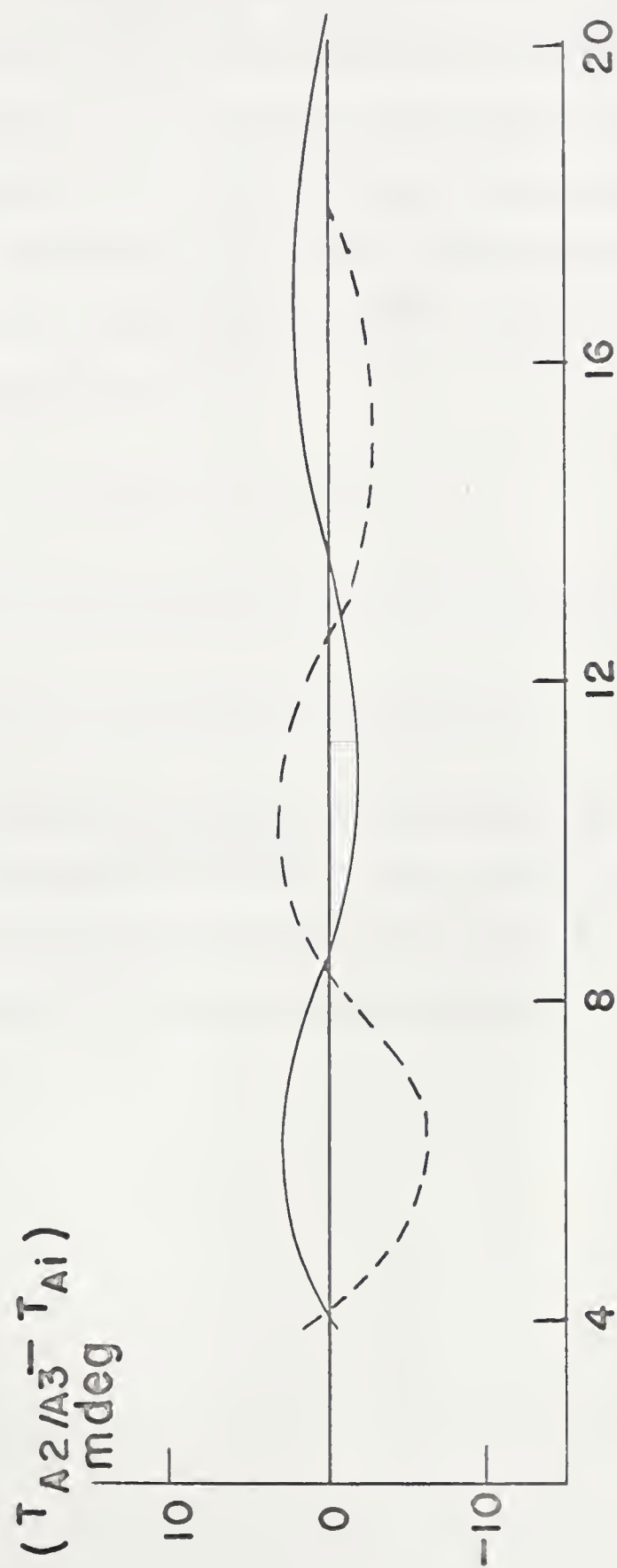
FIG. 5.5

For each set of coefficients the resistance values corresponding to temperatures ranging from 3° to 20°K at 10 millidegree intervals were tabulated by the computer. Using these three tables, temperatures $T_{A2/A3}$, T_{A2} and T_{A3} were obtained for given values of resistance in the range from 3° to 20°K . The temperatures T_{A2} and T_{A3} are found to lie within $\pm 0.1\%$ and $\pm 0.05\%$ respectively of the reference temperature, $T_{A2/A3}$. The deviation of T_{A2} (and T_{A3}) from $T_{A2/A3}$ has been plotted against $T_{A2/A3}$ in Fig. (5.6).

At liquid helium temperature the reproducibility of germanium thermometer CG-1 of Radiation Research Laboratory is ± 1.1 millideg on thermal cycling between liquid helium and room temperature. Martin (private communication) has worked with other thermometers of this type and found the reproducibility to be within ± 1 mdeg for some and as high as ± 5 mdeg for others. Thus it can be remarked that not all commercial germanium thermometers have high reproducibility. They should be tested over a period of time and frequent spot checks are necessary before the thermometer can be accepted as a secondary standard.

FIGURE 5.6
DEVIATION OF INTERPOLATED TEMPERATURES
 T_{A2} AND T_{A3} FROM $T_{A2/A3}$
(See text)

RUN A2 ---
RUN A3 —



TEMPERATURE A2/A3 °K

GE. THERMOMETER (CG-1)

5.3 Superconducting Transition Temperature of Lead

The superconducting transition temperature of 69 grade lead has been found against the calibrated 10 Ω carbon thermometer. The carbon thermometer was calibrated by gas thermometer at helium filling pressures of about 32 mm Hg and 77 mm Hg at 4°K. The corresponding transition temperatures are 7.195°K and 7.197°K. The average transition temperature is:

$$7.196^{\circ}\text{K} \pm 0.005$$

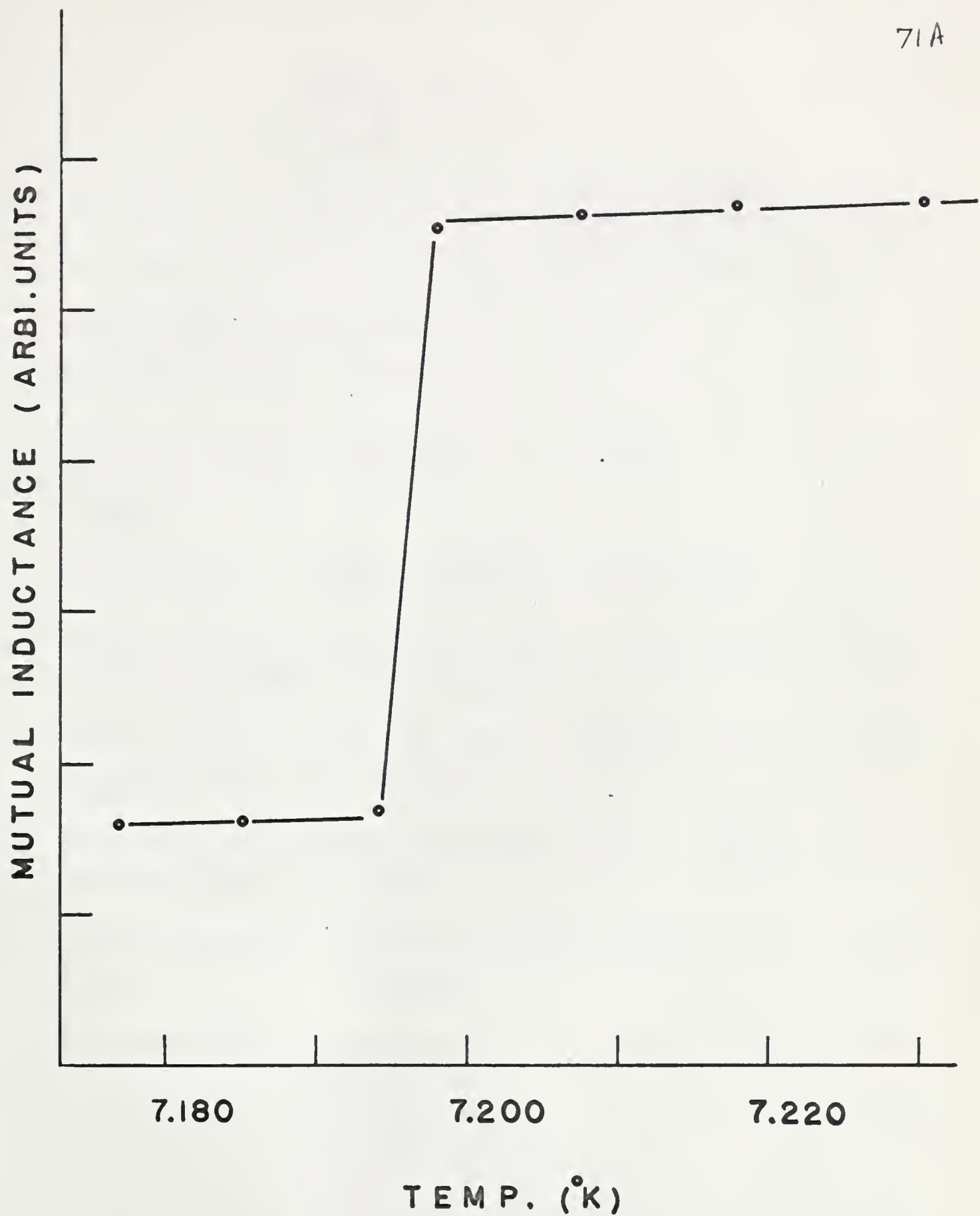
The superconducting transition curve is shown in Fig. (5.7).

$$\text{Width of transition} = 0.003^{\circ}\text{K}.$$

Table (5.3.1) gives the survey of previous determinations of transition temperature of lead. With the exception of the value given by Pearson and Templeton there is a satisfactory agreement between all the previous determinations within the error limits.

FIGURE 5.7

SUPERCONDUCTING TRANSITION IN LEAD



LEAD TRANSITION TEMP = $7.196 \pm 0.005^\circ\text{K}$

FIG. 5.7

TABLE 5.3.1

No.	Authors	Material	Superconducting transition temp- erature °K.	Width of transition °K.
1.	KamerlinghOnnes and Tuyn (1922)	Kahlbaum 99.99%	7.2	
2.	de Haas, de Boer and Van den Berg (1934).	Kahlbaum 99.99%	7.193	0.02
3.	Daunt (1939)	Hilger 99.99%	7.22 ± 0.03	
4.	Ziegler, Brucksch and Hickman(1942)	Extruded wires, Baker and Co.	7.20 ± 0.01	0.03
5.	Boorse, Cook & Zemansky(1950)	National Lead Co. 99.999%	7.224 ± 0.04	0.05
6.	Pearson & Templeton(1958)	Johnson-Matthey 99.999%	7.175 ± 0.005	0.001
7.	Franck and Martin (1961)	Johnson-Matthey 99.999%	7.193 ± 0.005	0.004
8.	This work(1966)	Consolidating Mining and Smelting Co, 99.9999% Isotopic composition lead 204: 206:207:208:: 1:16:15: 36.	7.196 ± 0.005	0.003

5.4 Estimation of Errors

The errors arising from the following sources were considered in calculating the absolute temperatures.

- (1) Vapour pressure measurement: A maximum error of 0.2mmHg appears in reading the vapour pressure manometer. This amounts to 0.3 mdeg at the normal boiling point of helium used as the calibration temperature. The 1958 ^4He scale of temperatures was used.
- (2) Gas pressure measurement: The precision of this measurement using a Wild Cathetometer KM268 is about 0.02mm. This corresponds to 1.5 mdeg at the lowest filling pressure (32mmHg at 4°K). At the highest filling pressure (100 mm Hg at 4°K) the error is 0.5 mdeg.
- (3) Correction for dead volume: An error of 15% in the term $\sum_i \frac{Pv_i}{T_i}$ due to errors in V_i and T_i determinations gives rise to an error of 0.2 mdeg at 4°K

4.0 mdeg at 20°K

 This error is independent of the filling pressure of gas thermometer.
- (4) Correction for non-ideality of gas: For a filling pressure of 32 mm Hg at 4°K , this correction amounts to 0.041°K and for the filling pressure of 77 mm Hg at 4°K to 0.092°K . Using the virial coefficients of Kilpatrick²³⁾ et al. for calculating this correction, the corresponding figures are 0.043°K and 0.097°K . These figures give the idea of the

possible uncertainty in this correction.

- (5) Correction for thermomolecular pressure difference: This correction is a fraction of a millidegree and has been neglected.

In view of the above estimation of errors, we assign a maximum error limit of ± 5 mdeg to the accuracy of measurement of the superconducting transition temperature of lead. This, however, does not include the uncertainty of about ± 2 mdeg in the 1958 ^4He vapour pressure scale of temperatures used for calibration of the gas thermometer.

CONCLUSIONS6.1 Thermometry Results

The reproducibility and sensitivity of germanium thermometer CG-1 of Radiation Research Laboratory are superior to carbon thermometer of Allen Bradley Co. The interpolation formula (cf. Eq. 2.24) for carbon thermometer gives temperature which is accurate to $\pm 0.7\%$ in the range 4° to 20°K . Temperatures calculated by the interpolation formula (cf. Eq. 5.1) for germanium thermometer are accurate to $\pm 0.4\%$ in this temperature range except in the vicinity of the point of inflection, (11.0°K), where the accuracy is 1.1% . These results are in agreement with the results of Clement and Quinell on Allen Bradley carbon thermometers and of Martin on germanium thermometers of this type.

6.2 Results for Lead

The superconducting transition temperature of high purity lead is $7.196^{\circ}\text{K} \pm 0.005^{\circ}$. This temperature can be used as a secondary fixed point.

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